U.S. DEPARTMENT OF COMMERCE
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PB-257 643

Translocation of Heptachlor and Chlordane from Indiana Cornfields

National Enforcement Investigations Center

Sep 75

265115

PB 257 643

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SPRINGFIELD, VA. 22161

BIBLIOGRAPHIC DATA SHEET	1. Report No. EPA/330/9-75/002	2.	3. Recipient's Accession No. PB-257 643
4. Title and Subtitle Translocation of Heptachlor and Chlordane from Indiana Cornfields.			5. Report Date September 1975 6.
7. Author(s)			8. Performing Organization Rept. No.
9. Performing Organization Name and Address National Enforcement Investigations Center Denver, Colorado			10. Project/Task/Work Unit No. 11. Contract/Grant No.
12. Sponsoring Organization Name and Address			13. Type of Report & Period Covered 14.

15. Supplementary Notes

16. Abstracts

Contents:

Description of the study area (Throckmorton farm, Sampling stations, Sampling schedule); Study findings (Chlordane, Heptachlor).

17. Key Words and Document Analysis. 17a. Descriptors

*Pesticides
*Chlorodan
*Heptachlor
Sampling
Farms
Wind direction
Wind velocity
Air pollution
Soils
Corn plants
Water pollution
Runoff
Field tests
Indiana

17b. Identifiers/Open-Ended Terms

17c. COSATI Field Group 6F, 2D

18. Availability Statement National Technical Information Service Springfield, VA 22161	19. Security Class (This Report) UNCLASSIFIED	21. No. of Pages 61
	20. Security Class (This Page) UNCLASSIFIED	22. Price PC$4.50 MF$2.25

NTIS-35 (REV. 10-73) ENDORSED BY ANSI AND UNESCO. THIS FORM MAY BE REPRODUCED USCOMM-DC 8265-P74

EPA-330/9-75-002

ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF ENFORCEMENT

TRANSLOCATION OF HEPTACHLOR AND CHLORDANE

FROM

INDIANA CORNFIELDS

September 1975

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
DENVER, COLORADO

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I. INTRODUCTION

In recent years, concern has been expressed by environmentalists and governmental agencies about the safety of the continued widespread use of the pesticides chlordane and heptachlor. After reviewing issues raised about environmental and health effects of these pesticides and their metabolites, the EPA Administrator issued a *Notice of Intent to Cancel Registration* of heptachlor and chlordane [Appendix A]. The notice was protested by manufacturers and formulators of these chlorinated hydrocarbon pesticides. As a result, the Office of General Counsel asked the National Enforcement Investigations Center (NEIC) to assist them in preparing evidential material in support of the hearing.

A literature review by NEIC scientists on air quality aspects of these two pesticides indicated, among other things, that the current applications of these pesticides allow their vapors and dusts to occur in ambient air masses. The Office of General Counsel then asked the National Enforcement Investigations Center to further document these literature survey findings with a verification study in an agricultural area, particularly one in which chlordane and heptachlor normally are used.

A field investigation was conducted from 24 April to 5 June 1975 during the corn planting season when the pesticides under study are normally disked into soil of cornfields as a preplanting farming practice.

The area selected for study was the Throckmorton Agricultural Research Center owned by Purdue University, West Lafayette, Ind. This farm was selected because 1) it is an applied agricultural research area; 2) the farm manager was willing to follow the NEIC study plan; and 3) the physical layout of the farm was appropriate for this study.

II. SUMMARY AND CONCLUSIONS

A field investigation was conducted from 24 April to 5 June 1975 to determine the fate of chlordane* and heptachlor which are disked into field soils before corn is planted. Two fields at the Purdue University Throckmorton Agricultural Research Center were selected for study. The smaller field, 12 ha (29 acres), was treated with a formulation of 72 ECF chlordane (72% technical chlordane) at a rate of 4.5 kg/ha (4 lb/acre). The larger field, 13 ha (32 acres), was treated with 3 ECF heptachlor (30.2% heptachlor) at the rate of 2.2 kg/ha (2 lb/acre). Immediately following soil treatment, corn was planted in each field.

Air samples were collected from 32 stations, 16 in each cornfield. Water samples from a tile drain underlying the heptachlor treated field, and surface runoff from both fields were collected and analyzed for pesticide contamination.

General Findings:

1. Two sampling techniques were used to determine translocation of heptachlor and chlordane into ambient air. One method used a nylon chiffon screen which was treated with ethylene glycol and suspended in a metal frame above the fields for approximately three days; the second method used an impinger-type sampler in which ambient air was drawn through a liquid reservoir of ethylene glycol for 24 hours. The aerial screen sampler was better for collecting the less volatile chlordane, and the impinger sampler was more efficient for collecting heptachlor.

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* *As used in this report, chlordane refers to "technical chlordane" which encompasses all its major isomers and related compounds.*

2. The study demonstrated that heptachlor and chlordane were translocated from soils upon which they were applied and contaminated the surrounding air and water environment.

3. Prevailing winds transported airborne heptachlor and chlordane compounds northeasterly across and away from the cornfields.

Specific Findings:

1. A preapplication sampling of the air above the two fields conducted two and one-half weeks before the pesticide application showed the presence of small amounts of chlordane, averaging 2.36 µg on the aerial screens and 5.5 ng/m^3 in the impinger sample. Heptachlor was not detected in any samples. A second sampling done just before application showed average chlordane levels of 0.37 µg on the aerial screens and 1.6 ng/m^3 in the impinger sample. Heptachlor was also found at low levels; aerial screens contained an average of 0.08 µg, with 0.94 ng/m^3 found in the impinger samples.

2. Ambient air sampling immediately after application of chlordane to the cornfield showed average chlordane levels of 22.1 µg on aerial screens and 30.5 ng/m^3 in impinger samples. Ambient air samples collected over the heptachlor treated field immediately after the pesticide application showed average levels of heptachlor were 0.51 µg on aerial screens and 244 ng/m^3 in impinger samples.

3. A second sampling of the air above the two pesticide treated cornfields approximately one week after application showed chlordane averaged 23.8 µg on the aerial screens and 13.9 ng/m^3 in the impinger samples. Heptachlor measured an average of 0.57 µg on aerial screens and 55.1 ng/m^3 in impinger samples.

4. A final sampling of the air about three weeks after pesticides were applied to the cornfields showed an average chlordane level of 7.19 µg on aerial screens and 16.9 ng/m^3 in the impinger samples. Heptachlor levels decreased to an average of 0.22 µg on aerial screens and 15.4 ng/m^3 in the impinger samples.

5. Rainfall and water from tile drains were collected prior to the application of pesticides to the cornfields. No heptachlor or chlordane were detected in these preapplication water samples. Following rainfalls, postapplication sampling of runoff water was done also. Surface runoff was collected from the heptachlor and chlordane treated cornfields. Analysis of a water sample collected on 22 May 1975 showed that runoff from the heptachlor treated field contained 152 µg/l of heptachlor. Runoff collected 30 May 1975 from the chlordane treated field contained 25 µg/l chlordane. Also, on 30 May 1975 a water sample from a tile drain underlying the heptachlor treated field was contaminated with 2.3 µg/l heptachlor.

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III. DESCRIPTION OF THE STUDY AREA

THROCKMORTON FARM

Field studies on the translocation of heptachlor and chlordane were conducted at the Throckmorton Agricultural Research Center in Tippecanoe County, Indiana. The farm was given to Purdue University in 1935 and has since been used as an agricultural research center and experimental station. It is located on State Highway 43 and county road 800 South, approximately 9 km (5-1/2 mi) southwest of Lafayette, Ind. [Fig. 1]. Of the 120 ha (297 acres) comprising the farm, 54 ha (134 acres) are in Section 5 (T 21 NR4W) and 66 ha (163 acres) in Section 6 (T 21 NR4W). The study area consisted of 13 ha (32 acres) in Section 5 where heptachlor was applied and 12 ha (29 acres) in Section 6 where chlordane was applied. The study site and surrounding acreage are characterized by gently rolling terrain with several drainage areas. Five watersheds are located on the chlordane field and four on the heptachlor field. The heptachlor field has a subsurface tile drain system which empties into a creek 75 m (246 ft) southeast of the heptachlor field [Fig. 2].

The chlordane field is bounded on the north and south by cornfields of 11 ha (27 acres) and 26 ha (65 acres), respectively. On the west is a 6.5 ha (16 acre) field of soybeans; Highway 43 and the heptachlor field are adjacent on the east.

Pasture land lies on the east and south boundaries of the heptachlor field. County road 800S borders on the north side of the field. Several residences are directly across the road, and numerous other residential areas are in the vicinity of the farm [Fig. 1]. In 1969 the present heptachlor field was sprayed with heptachlor; both fields were

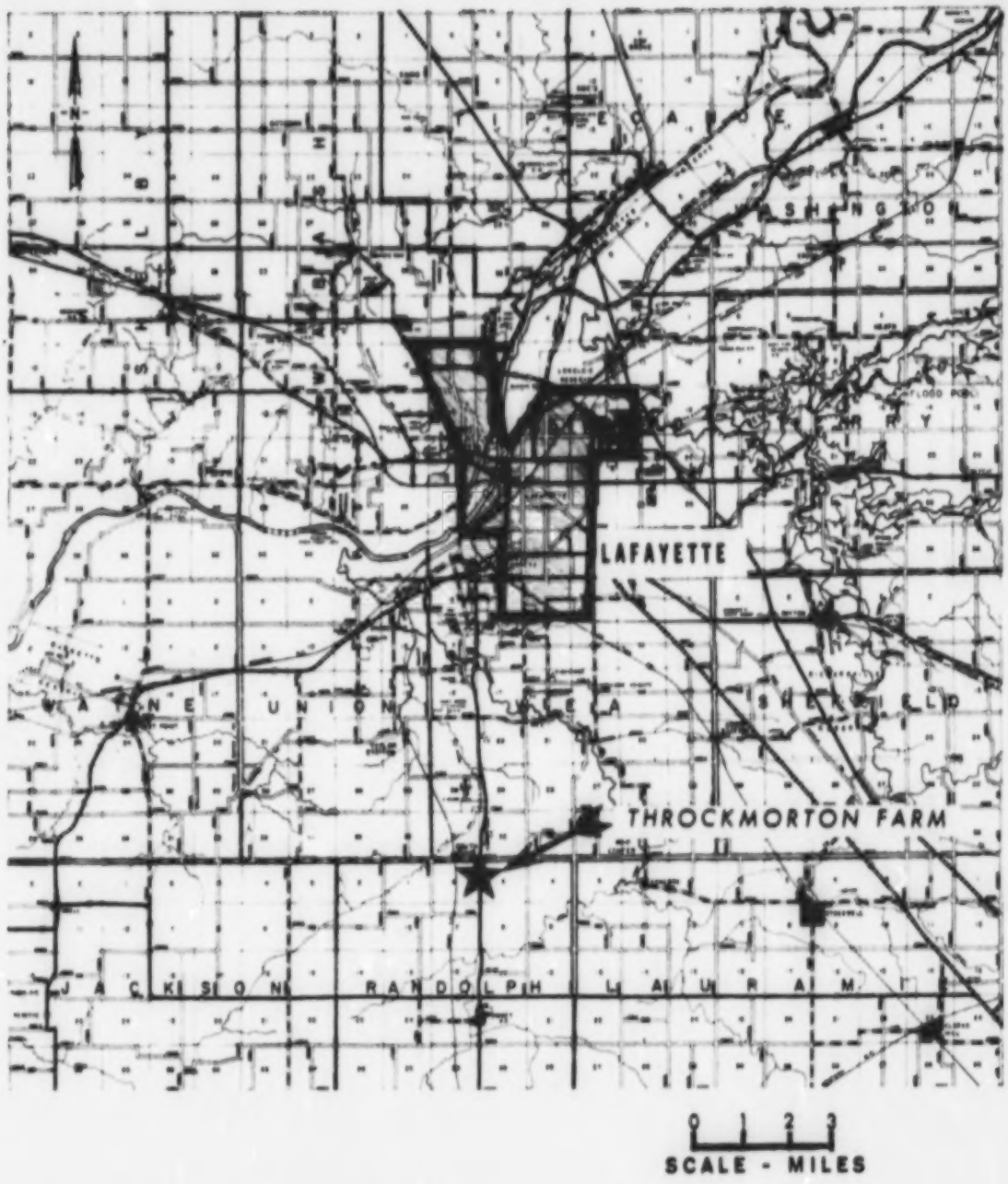

Figure 1. Location of Throckmorton Agricultural Research Center Tippecanoe County, Indiana

treated with aldrin in 1971. No pesticides have been applied to either field in the past four years.

SAMPLING STATIONS

Thirty-two sampling stations were established for this pesticide study, sixteen in each cornfield [Fig. 2].

Air samples were collected from each of these sampling locations by an aerial screen or an impinger-type air sampling apparatus. Descriptions of the sampling equipment are presented in Appendix B.

Square nylon chiffon screens (0.25 m^2; 2.25 ft^2) were suspended vertically from eleven wood-aluminum frames in the chlordane field. Two frames held a lower and upper screen approximately 1 and 2 m (3 and 6 ft) above the ground, while the remaining eight frames held single screens about 2 m above the ground.

Historical records indicated the prevailing winds were from the southwest; therefore, 1 double screen and 3 single screens were placed diagonally in the northeast corner of the field. A double screen was also placed in the center of the field facing southwest into the prevailing winds. Single screens were placed in the northwest, southeast and center of the field in the event that wind direction changed.

The placement of the frames in the heptachlor field was similar to that of the chlordane field. These frame locations were used during each phase of the study.

Air sampling units (ethylene glycol impinger apparatus) were periodically operated at three locations in each cornfield. The samplers were placed near the center and in the northeast and southwest corners of the heptachlor- and chlordane-treated fields [Fig 2].

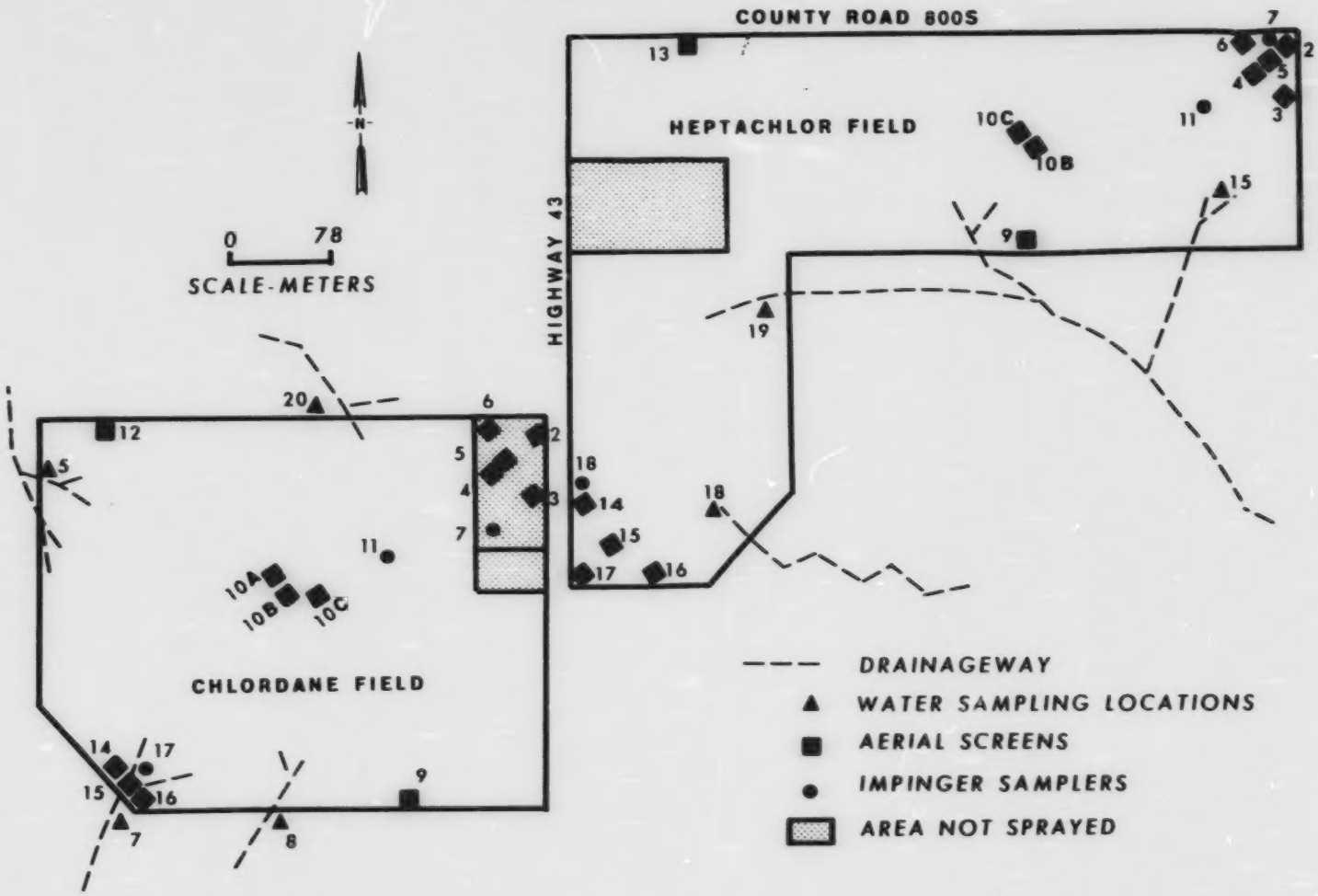

Figure 2. Sampling Locations Throckmorton Agricultural Research Center

Tippecanoe County, Indiana

SAMPLING SCHEDULE

Air sampling began in April 1975 to correspond with the corn planting season and the normal time for application of heptachlor and chlordane. The study consisted of five sampling sequences, four days each. Sequences I and II occurred before application of the pesticides to obtain background data. Sequence III began immediately after application, sequence IV was 6 to 7 days after application, and sequence V was 20 to 21 days after application [Table 1]. Analytical methods and results, as well as chain of custody procedures followed during the survey are described in Appendices C, D and E, respectively.

Table 1

SAMPLING SCHEDULE
THROCKMORTON AGRICULTURAL RESEARCH CENTER
TIPPECANOE COUNTY, INDIANA
24 April-5 June 1975

Sequence	Date (1975)	Sample	Sampling Method	Exposure Time (hours)
I-Preapplication	4/24-27	Air	Aerial Screens	48
		Air	Impinger	24
II-Preapplication	5/8-12	Air	Aerial Screen	85
		Air	Impinger	24
		Rainfall	Composite	2
		Tile Drain	Grab	-
III-Postapplication	5/12-16	Air	Aerial Screens	68
		Air	Impinger	24
IV-Postapplication	5/19-22	Air	Aerial Screens	70
		Air	Impinger	24
		Surface Runoff	Grab	-
		Tile Drain	Grab	-
	5/24	Tile Drain	Grab	-
	5/30	Tile Drain	Grab	-
		Surface Runoff	Grab	-
V-Postapplication	6/2-5	Air	Aerial Screens	70
		Air	Impinger	24
		Tile Drain	Grab	-

IV. STUDY FINDINGS

CHLORDANE

Field investigations began approximately two and one-half weeks before chlordane was applied to the cornfield in the study area. Samples of ambient air contaminants were collected at 14 locations [Appendix D, Table D-6]. Analyses of these air samples revealed the presence of technical chlordane. Square aerial screens (0.25 m^2; 2.25 ft^2) exposed to the ambient air for 48 hours collected an average of 2.36 µg chlordane. An impinger-type sampler operating 24 hours in the southwest corner of the field collected 10.9 ng/m^3 of technical chlordane [Table D-6]. Impinger-type samplers operating the same period in the center and northeast corner of the field measured no technical chlordane. Air contaminants were sampled a second time, just before chlordane was applied to the cornfield. Aerial screens set up in the cornfield for 85 hours collected an average level of 0.37 µg technical chlordane. Once again the impinger-type sampler operated 24 hours in the southwest corner of the field, collecting 3.2 ng/m^3 technical chlordane, while the other units measured no chlordane in the ambient air near the center and northeast corner of the field.

The only other sample collected from this cornfield during the preapplication survey was a 2-hour composite rainwater sample. Analyses revealed that the water sample was not contaminated with chlordane.

Chlordane detected during the preapplication survey was presumed to have come from surrounding areas where it is used in both household and agricultural pesticide applications. Specific sources of this background contamination were not determined.

On 13 May 1975 a formulation of 72 ECF Chlordane (72% technical chlordane) was applied at a rate of 4.5 kg/ha (4 lb/acre) by a tractor mounted sprayer and disked into the cornfield soil. Immediately after the pesticide application, aerial screens and impinger air samplers were set up at the predetermined sampling stations in the cornfield.

Analyses of samples collected by these devices revealed that chlordane translocated from the cultivated soils into ambient air immediately after application. Aerial screens exposed to the ambient air over the cornfield immediately after application for a duration of 68 hours collected an average level of 22.1 μg technical chlordane. This represents an increase in technical chlordane on the aerial screens when compared with preapplication levels [Fig. 3].

Air samples collected by all three impinger samples contained technical chlordane also. Sampling units operating in the northeast and southwest corners of the field for 24 hours, immediately following chlordane application, collected 37.9 and 23.1 ng/m^3 of technical chlordane. The impinger sampler operating in the center of the field collected 0.20 μg of technical chlordane; however, operating time of this unit was not recorded because of a malfunction in the timer.

Figure 3 shows a general pattern of chlordane translocation across the cornfield from southwest to northeast. During the first 24 hours of the sampling period (13-14 May 1975) prevailing winds of 6 to 10 knots (7 to 12 mph) blew across the field in this same direction, southwest to northeast [Fig. 4]. Although many other factors including wind velocity, soil binding properties, humidity, vapor pressures and cultivation practices may have influenced translocation, these observed wind conditions correlated with the progressive increase in chlordane contamination across the cornfield.

Figure 3. Relative Abundance of Chlordane in Ambient Air Samples at an Indiana Cornfield April-June 1975 (Aerial Screens)

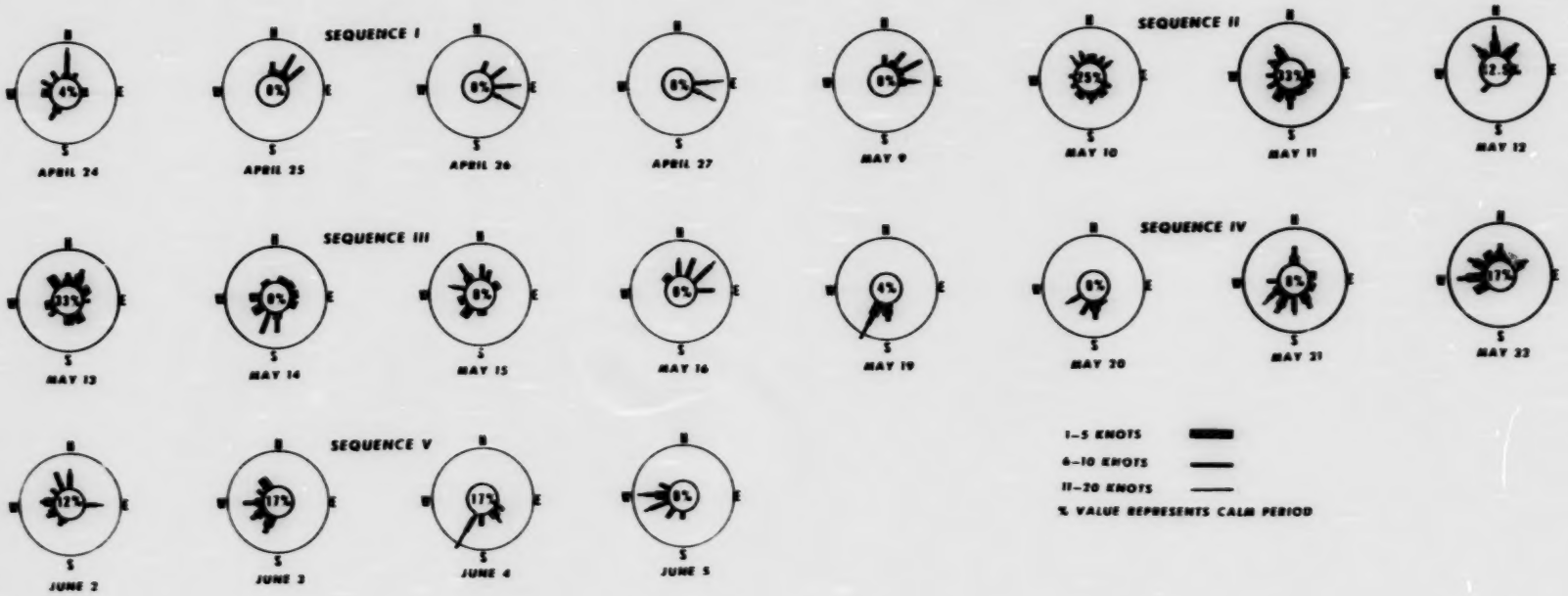

Figure 4. Wind Direction And Velocity, Lafayette, Indiana

April–June,1975

Six days after application of chlordane on the cornfield, another series of air samples was collected. Aerial screens were exposed to the ambient air for 70 hours. Analyses showed amounts of technical chlordane in the air ranging from 6.4 µg in the southwest corner of the field to 43.0 µg in the northeast corner. The pattern of chlordane distribution was again from the southwest to the northeast, corresponding with prevailing winds [Figs. 3, 4].

Impinger collected air samples showed the persistence of technical chlordane in the air also. Sampling units, operated for 24 hours in the northeast and southwest corners of the cornfield, collected air containing 13.7 and 14.1 ng/m^3 of technical chlordane. The impinger in the center of the field (with a faulty timer) collected 0.12 µg of technical chlordane.

A series of water samples was collected from drainageways in watersheds 5, 7 and 8 in the chlordane treated cornfield during this segment of the survey [Fig. 2]. These surface water samples contained chlordane levels ranging from 0.3 to 1.6 µg/l, indicating that the pesticide was being washed from field soils following local rain showers.

Twenty days after application of chlordane on the cornfield, a final series of air contaminant samples was collected. After 70 hours of exposure to ambient air of the cornfield, aerial screens were analyzed for chlordane contamination. Analyses of the screen contaminants showed a decline in technical chlordane to an average of 7.19 µg.

Air contaminants collected by the three impinger samplers during a 24 hour period showed the center of the field and the northeast corner contained 0.92 µg and 33.8 ng/m^3 of technical chlordane, respectively. Ambient air drawn through the impinger unit in the southwest corner of the field contained no chlordane. Distribution patterns of chlordane across the cornfield were again generally in the direction of the prevailing wind, southwest to northeast [Figs. 3, 4].

Following rainfalls, another postapplication sampling of water was done. Surface runoff was collected from drainageways in watersheds 5, 7 and 8 in the chlordane treated cornfield. The three samples contained 1.6, 0.3, and 0.8 µg/l of technical chlordane, respectively.

This study has demonstrated that chlordane was translocated from soils upon which it has been applied into water supplies and into ambient air masses.

HEPTACHLOR

A second field investigation was conducted on the same farm in an adjacent cornfield to the northeast of the chlordane treated field [Fig. 2]. The purpose of this study was to determine the fate of heptachlor disked into field soil prior to planting corn. The study sampling periods and pesticide application generally corresponded with those described for the chlordane study.

Samples of ambient air contaminants were collected on 26 April and 12 May 1975 to determine levels of heptachlor prior to treating the soils of the study area cornfield with the pesticide. No heptachlor was detected in the ambient air in April, while low levels of 0.07 and 0.12 µg heptachlor on screens and 0.94 ng/m^3 in impinger samples were recorded in May.

Rainfall and water from tile drains underlying the cornfield were collected prior to the application of heptachlor to field soils. No contamination by heptachlor was detected in these water samples.

Heptachlor detected in air samples during the preapplication survey was presumed to have come from surrounding areas, but specific sources were not determined.

On 12 May 1975, a formulation of 3 ECF heptachlor (30.2% heptachlor) was applied at the rate of 2.2 kg/ha (2 lb/acre) by a tractor mounted sprayer and disked into the cornfield soil. Immediately after application, as well as 7 and 21 days later, ambient air was sampled at various locations in the heptachlor treated cornfield. Samples of contaminants, collected by aerial screens exposed to the air masses flowing over the cornfield, generally showed a peak increase in heptachlor contamination 7 days after the pesticide was applied to the cornfield soil [Table D-4 and Fig. 5]. Typically, during all postapplication sampling periods, the highest values for heptachlor were recorded from screens set up in the northeastern section of this L-shaped cornfield which corresponded with the direction of the prevailing winds.

Airborne heptachlor collected by impinger samplers during the postapplication studies progressively decreased with time [Tables D 3-5]. Nevertheless, the highest values were recorded from the sampling units in the northeastern part of the cornfield. Distribution patterns of air-carried heptachlor were southwest to northeast, again corresponding with prevailing wind direction.

Analyses of samples collected on 22 and 30 May 1975 showed that rainwater runoff from the cornfield contained as much as 152 µg/l of heptachlor. Additionally, a water sample from the tile drain collected on 30 May 1975 showed the subsurface water system contained 2.3 µg/l of heptachlor.

This study has demonstrated that, after rainfalls, heptachlor was washed from and percolated through soils into surface and ground water systems. Furthermore, the study demonstrated that measurable quantities of heptachlor were translocated from cornfield soils into ambient air masses flowing over the fields.

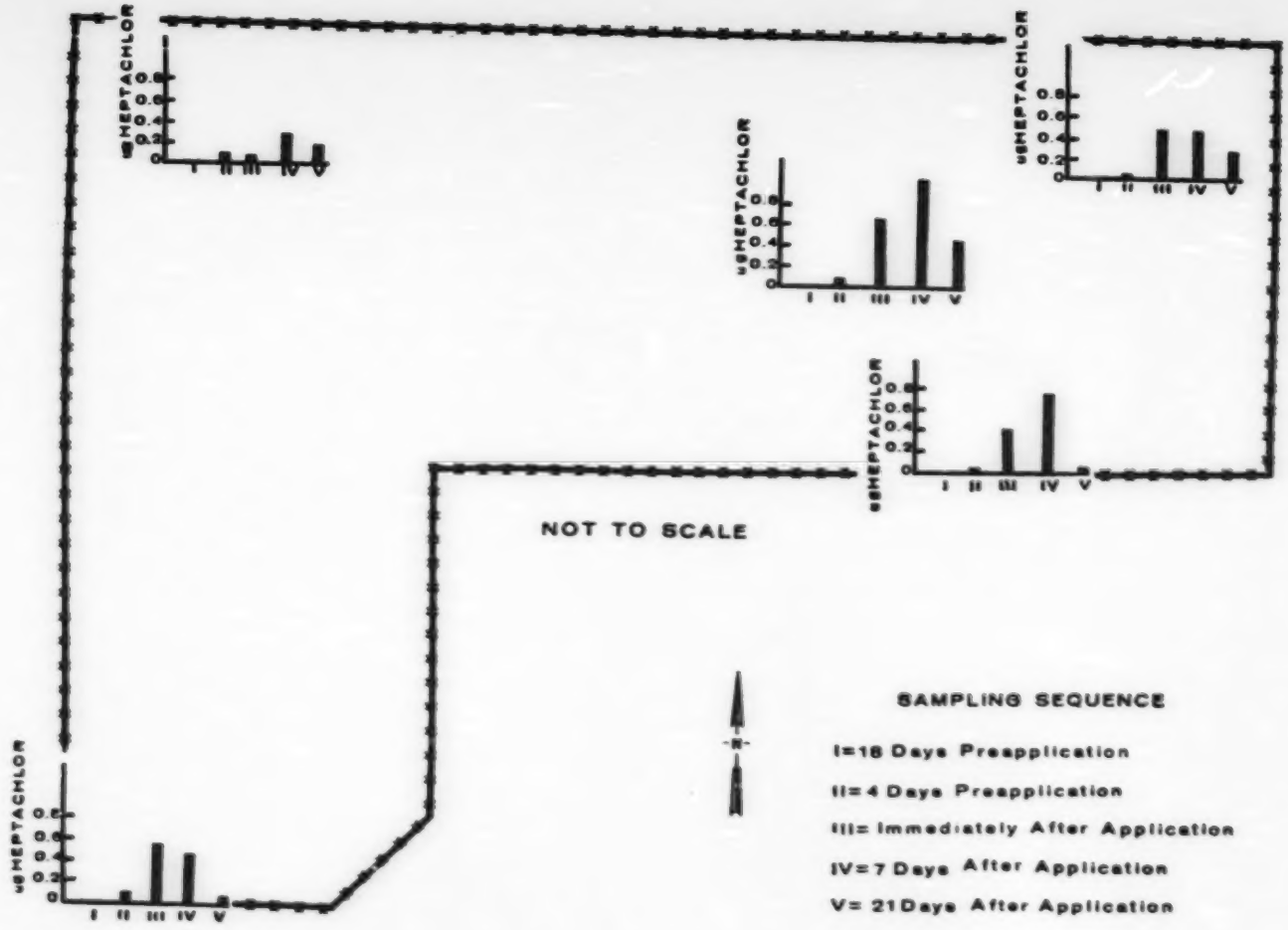

Figure 5. Relative Abundance of Heptachlor in Ambient Air Samples at an Indiana Cornfield April-June 1975 (Aerial Screens)

APPENDIX A
CORRESPONDENCE

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UNITED STATES OF AMERICA

ENVIRONMENTAL PROTECTION AGENCY

NOTICE OF INTENT TO CANCEL REGISTRATIONS
HEPTACHLOR OR CHLORDANE

On March 18, 1971, the Administrator of this Agency announced that active internal review was being initiated on a number of pesticide products, including those containing chlordane and heptachlor. As the result of such review and for the reasons set forth in the attached Statement of Reasons, I find that the continued registration and use of these pesticides appear to pose substantial questions of safety amounting to an unreasonable risk to man and the environment. I therefore serve and file this Notice of Intent, together with the attached Statement of Reasons, to cancel all registered uses of heptachlor and chlordane within thirty (30) days, pursuant to Section 6 of the Federal Insecticide, Fungicide, and Rodenticide Act, as amended, (86 Stat. 973, 7 U.S. § 136d), with the exception of the use of heptachlor or chlordane through subsurface ground insertion for termite control and the dipping of roots or tops of nonfood plants. Any affected party may contest this action by requesting a hearing on specific registered uses within thirty (30) days of the date of this notice. Requests for hearings should be submitted to the Agency's hearing clerk at the following address:

Mrs. Betty J. Billings
Hearing Clerk
U.S. Environmental Protection Agency
Room 1019, Waterside Mall - East Tower
401 M Street, S.W.
Washington, D.C. 20460

The proposed cancellation shall become final and effective thirty (30) days from the date of this notice as to those registered uses for which a hearing is not requested by any affected party. The proposed cancellation shall not take effect regarding any registered use for which a hearing is requested until the hearing has been completed, unless there is a concurrence from all parties to the proceeding. The Agency reserves the opportunity to present evidence on any registered use affected by this order regardless of whether or not a hearing has been requested on that use, or whether or not such use is to be actively defended in the hearings.

Russell E. Train
Administrator

Dated: Nov 18, 1974

STATEMENT OF REASONS: Heptachlor and Chlordane

I. Legal Authority

Section 6 (b) of the Federal Insecticide, Fungicide, and Rodenticide Act (7 U.S.C. § 135 et seq) as amended (7 U.S.C. § 136a(c)(5)(D)) authorizes the Administrator of the Environmental Protection Agency (or his designee) to issue a notice of intent to cancel the registration of a pesticide or to hold a hearing "[i]f it appears to the Administrator that a pesticide or its labeling . . . does not comply with the provisions of this act or, when used in accordance with widespread and commonly recognized practice, generally causes unreasonable adverse effects on the environment. . . ." The phrase "unreasonable adverse effects on the environment" is defined in Section 3 of the Act (7 U.S.C. § 136(bb)) as "any unreasonable risk to man or the environment taking into account the economic, social, and environmental costs and benefits of any pesticide."

The Act also prohibits the sale of pesticides which are misbranded. A product is considered misbranded if the label does not contain directions for use and a warning or caution statement which are necessary and if complied with are adequate to protect health and the environment. (7 U.S.C. § 136(a)(1)(F) and (G)).

APPENDIX B

SAMPLING EQUIPMENT AND METHODS

SAMPLING EQUIPMENT AND METHODS

AIR SAMPLER

The air-sampling unit is shown schematically in Fig. B-1. It contains a pump that draws air through a sampling series or train and auxiliary equipment to control and measure air flow, switching the flow from one sampling train to another after a pre-set interval.

(a) Sampling train- Each sampling train contains an impinger containing 100 ml of ethylene glycol to trap particulates and gasses and an adsorption tube packed with a plug of glass wool to remove any splashover or water condensation.

(b) Flow control and pump - After the air has been pulled through the sampling train, it passes through a solenoid valve controlled by a timer, through a control valve by which the air flow in the sampling train may be set at the desired rate as shown on a flowmeter, through the flowmeter, and finally through the vacuum pump. A momentary contact switch may be closed to switch the air flow to a particular sampling train so that its flow may be adjusted or read any time.

Three air sampling units were used in each field. Each unit was given a station number and was used only at that station. The impingers were filled with approximately 100 ml of ethylene glycol. Flow rates were set for 0.9 standard ft^3/min. The timers were adjusted to permit collection of two samples in 24 hours (the dial calibrated in percent of a 24-hour day). Pesticide-free aluminum foil was used to cover the air intake tubes while transporting the units to their sampling locations in the fields.

A gas generator provided the power supply at station B-17. The generator was set approximately 23 m (75 ft) away to avoid exhaust contamination.

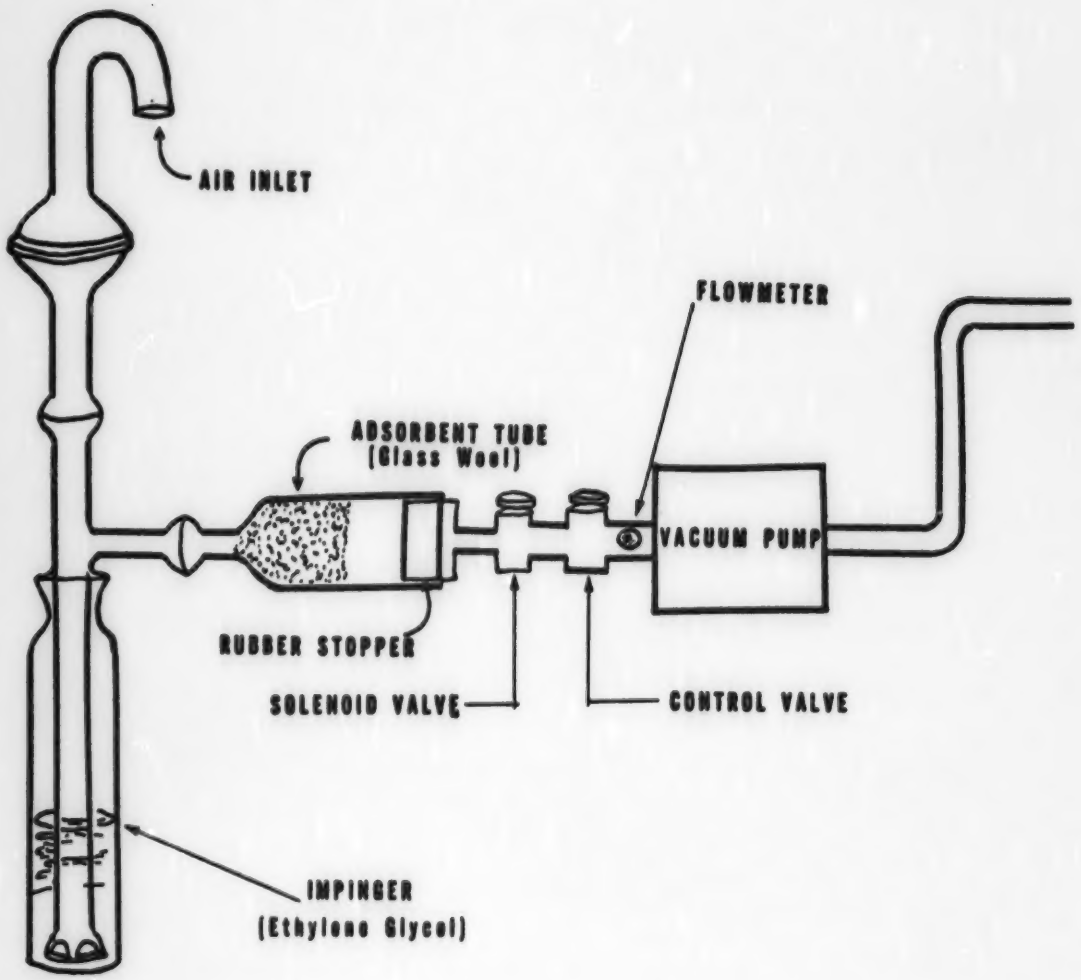

Figure B–1. Schematic Drawing of Impinger-Type Air Sampling Unit

The sampling units shutoff automatically after 24 hours. The units were then transported to the servicing area and disassembled. The ethylene glycol was removed from the sampling trains and transferred to 250 ml screw-cap glass bottles. Impingers were rinsed with approximately 25 ml ethlene glycol which was added to the samples. The bottles were appropriately labeled and stored in an ice chest for transportation to the laboratory under chain of custody procedures [Appendix E].

The ethylene glycol was replaced in the impingers, fresh glass wool plugs were inserted, the timers were reset, and pesticide free aluminum foil was placed on the ends of the air intake tubes. The samplers were then stored until the next sampling sequence.

AERIAL SCREEN APPARATUS

Each aerial screen apparatus consisted of a wood and aluminum framework. Two 10-ft poles were attached to a 76 x 101 cm (30 x 40 in) aluminum channel frame, and each apparatus was placed at a sampling site in the cornfield study area. The top of each frame was above ground level.

Pretreated nylon chiffon cloths (aerial screens) 0.5 m^2 were suspended vertically within the aluminum frame. These aerial screens were securely suspended in the framework to ensure that they would withstand sudden gusts of wind and not be blown from the frames. Screens used in this study were prepared by the method of Tessari and Spencer.* This method employs treating the screens with a 10% ethylene glycol and acetone solution. All screens were prepared at NEIC-Denver and transported to the study site in chemically clean glass jars.

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* *Tessari, J. D. and D. L. Spencer, 1971. Air sampling in the Human Environment. Jour. AOAC, 54 (6): 1376-1382.*

Rubber gloves, pre-rinsed in ethylene glycol, were worn while the screens were suspended within the frame. A different pair of gloves was worn in each field. At the end of each exposure, the screens were removed (again using pre-rinsed rubber gloves), placed in chemically clean glass jars, packed in a shipping container, and transported to the laboratory under chain of custody procedures [Appendix E].

APPENDIX C

ANALYTICAL METHODS

ANALYTICAL METHODS

EXTRACTION

1. Aerial Screen Samples: The nylon chiffon screens were extracted in small soxhlet extractors for 4 hr with 150 ml of 1:1 hexane and acetone. The solvent extract was then back extracted with 250 ml water in a 500 ml separatory funnel. The solvent layer was removed and the water extracted again with two 75 ml portions of hexane. The combined solvent was concentrated to 10 ml on a hot water bath under a gentle stream of filtered air.

2. Impinger Samples: Ethylene glycol samples (ca 250 ml) were extracted in a 500 ml separatory funnel with two 75 ml portions of hexane. The combined hexane extract was then back extracted with 250 ml water in the separatory funnel, the solvent layer removed and the water again extracted with two 75 ml portions of hexane. The combined solvents were then concentrated to 10 ml on the hot water bath.

3. Water samples (ca 1 liter) were serially extracted in a 2,000 ml separatory funnel with two 150 ml portions of hexane. When difficult emulsions were encountered, the samples were extracted again with a third 150 ml portion of hexane. The combined hexane extracts were then concentrated to 10 ml on a hot water bath under a gentle stream of filtered air.

ALUMINA CLEANUP

Both the cloth and the ethylene glycol extracts still contained gross amounts of interfering compounds after back extraction with water and required additional cleanup. The extracts were added to 2 x 15 cm columns of activated alumina deactivated with 3% water and prewetted with hexane.* The columns were eluted with 10% ethyl ether in hexane

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* *Boyle, W. W., R. H. Burttschell and A. A. Rosen. Infrared Fish. Advances in Chemistry Series, No. 60: Organic Environment.*

and four 50 ml fractions were collected. Heptachlor elutes almost entirely in fraction 1, trans-nonachlor in fraction 2, and gamma and alpha chlordane in fractions 3 and 4. Fractions 3 and 4 are combined for subsequent electron-capture gas chromatography. The fractions were again concentrated to 10 ml on the hot water bath.

ELECTRON-CAPTURE GAS CHROMATOGRAPHY

A 1 µl aliquot of the alumina column fractions of each extract was analyzed by electron-capture gas chromatography. Minimum detectable limits for heptachlor and chlordane were 0.01 to 0.1 µg, respectively. Quantitative estimates were made by computer calculation after calibration runs on standard insecticides run at the same concentration levels as the samples. The following gas chromatographic conditions were used:

Gas Chromatograph	Hewlett/Packard Model 5713 with Automatic Sampler
Detector	Electron-Capture Ni-63
Column	2 mm ID x 6' - Glass 6% OV-101 on 60/80 GC-Q
Carrier Gas	P-10 (90% Argon/10% Methane)
Flow	20 ml/min
Temperatures:	
Injector	200°C
Column	200°C
Detector	250°C

APPENDIX D
ANALYTICAL RESULTS

Tables D 1-11

ANALYSES OF AMBIENT AIR AND WATER CONTAMINANTS FROM AN INDIANA CORNFIELD

Sample	Location	Date (1975)	Type	Results
	1. Air analyses. Field to be treated with heptachlor. Sequence 1, preapplication.			
H-7	NE corner	4/28	Impinger	ND†
H-11	Center	4/26	Impinger	ND
H-18	SW corner	4/25	Impinger	ND
H-2	NE corner	4/26	Screen	ND
H-3	NE corner	4/26	Screen	ND
H-4	NE corner	4/26	Screen	ND
H-5	NE corner	4/26	Screen	ND
H-6	NE corner	4/26	Screen	ND
H-9	SE corner	4/26	Screen	ND
H-10	Center	4/26	Screen	ND
H-13	NW corner	4/26	Screen	ND
H-14	SW corner	4/26	Screen	ND
H-15	SW corner	4/26	Screen	ND
H-16	SW corner	4/26	Screen	ND
H-17	SW corner	4/26	Screen	ND
	2. Air analyses. Field to be treated with heptachlor. Sequence 2, preapplication.			
H-7	NE corner	5/11	Impinger	ND
H-11	Center	5/11	Impinger	0.908 ng/m^3
H-18	SW corner	5/11	Impinger	0.962 ng/m^3
H-2	NE corner	5/12	Screen	0.06 total µg
H-3	NE corner	5/12	Screen	0.06 " "
H-5	NE corner	5/12	Screen	0.08 " "
H-6	NE corner	5/12	Screen	0.09 " "
H-9	SE corner	5/12	Screen	0.04 " "
H-10-B	Center	5/12	Screen	0.05 " "
H-10-C	Center	5/12	Screen	0.09 " "
H-13	NW corner	5/12	Screen	0.12 " "
H-14	SW corner	5/12	Screen	0.10 " "
H-15	SW corner	5/12	Screen	0.08 " "
H-16	SW corner	5/12	Screen	0.13 " "
H-17	SW corner	5/12	Screen	0.11 " "

† *Not detected*

Tables D 1-11 (Cont.)

ANALYSES OF AMBIENT AIR AND WATER CONTAMINANTS FROM AN INDIANA CORNFIELD

Sample	Location	Date (1975)	Type	Results
	3. Air analyses. Field treated with heptachlor. Sequence 3, postapplication			
H-7	NE corner	5/13	Impinger	145 ng/m^3
H-11	Center	5/13	Impinger	474 "
H-18	SW corner	5/13	Impinger	112 "
H-2	NE corner	5/15	Screen	0.51 total µg
H-3	NE corner	5/15	Screen	0.61 " "
H-4	NE corner	5/15	Screen	0.44 " "
H-5	NE corner	5/15	Screen	0.52 " "
H-6	NE corner	5/15	Screen	0.47 " "
H-9	SE corner	5/15	Screen	0.42 " "
H-10-B	Center	5/15	Screen	0.64 " "
H-10-C	Center	5/15	Screen	0.75 " "
H-13	NW corner	5/15	Screen	0.10 " "
H-14	SW corner	5/15	Screen	0.59 " "
H-15	SW corner	5/15	Screen	0.46 " "
H-16	SW corner	5/15	Screen	0.46 " "
H-17	SW corner	5/15	Screen	0.71 " "
	4. Air analyses. Field treated with heptachlor. Sequence 4, postapplication			
H-7	NE corner	5/21	Impinger	47.8 ng/m^3
H-11	Center	5/21	Impinger	80.2 "
H-18	SW corner	5/21	Impinger	37.2 "
H-2	NE corner	5/22	Screen	0.45 total µg
H-3	NE corner	5/22	Screen	0.86 " "
H-4	NE corner	5/22	Screen	0.33 " "
H-5	NE corner	5/22	Screen	0.63 " "
H-6	NE corner	5/22	Screen	0.24 " "
H-9	SE corner	5/22	Screen	0.75 " "
H-10-B	Center	5/22	Screen	0.94 " "
A-10-C	Center	5/22	Screen	1.08 " "
H-13	NW corner	5/22	Screen	0.28 " "
H-14	SW corner	5/22	Screen	0.29 " "
H-15	SW corner	5/22	Screen	0.57 " "
H-16	SW corner	5/22	Screen	0.66 " "
H-17	SW corner	5/22	Screen	0.30 " "

Tables D 1-11 (Cont.)

ANALYSES OF AMBIENT AIR AND WATER CONTAMINANTS FROM AN INDIANA CORNFIELD

Sample	Location	Date (1975)	Type	Results
	5. Air analyses. Field treated with heptachlor. Sequence 5, postapplication			
H-7	NE corner	6/4	Impinger	20.6 ng/m^3
H-11	Center	6/4	Impinger	20.0 "
H-18	SW corner	6/4	Impinger	5.77 "
H-2	NE corner	6/5	Screen	0.29 total µg
H-3	NE corner	6/5	Screen	††
H-4	NE corner	6/5	Screen	0.26 " "
H-5	NE corner	6/5	Screen	0.39 " "
H-6	NE corner	6/5	Screen	0.33 " "
H-9	SE corner	6/5	Screen	0.07 " "
H-10-B	Center	6/5	Screen	0.45 " "
H-10-C	Center	6/5	Screen	0.41 " "
H-13	NW corner	6/5	Screen	0.19 " "
H-14	SW corner	6/5	Screen	0.07 " "
H-15	SW corner	6/5	Screen	0.13 " "
H-16	SW corner	6/5	Screen	0.03 " "
H-17	SW corner	6/5	Screen	0.05 " "
	6. Air analyses. Field to be treated with chlordane. Sequence 1, preapplication			
B-7	NE corner	4/26	Impinger	ND
B-11	Center	4/26	Impinger	ND
B-17	SW corner	4/25	Impinger	10.9 ng/m^3
3-2	NE corner	4/26	Screen	3.48 total µg
B-3	NE corner	4/26	Screen	3.62 " "
B-4	NE corner	4/26	Screen	2.91 " "
B-5	NE corner	4/26	Screen	3.48 " "
B-6	NE corner	4/26	Screen	2.40 " "
B-9	SE corner	4/26	Screen	2.08 " "
B-10	Center	4/26	Screen	1.70 " "
B-12	NW corner	4/26	Screen	1.55 " "
B-14	SW corner	4/26	Screen	1.38 " "
B-15	SW corner	4/26	Screen	1.02 " "
B-16	SW corner	4/26	Screen	2.40 " "

†† *Sample spilled and lost*

Tables D 1-11 (Cont.)

ANALYSES OF AMBIENT AIR AND WATER CONTAMINANTS FROM AN INDIANA CORNFIELD

Sample	Location	Date (1975)	Type	Results
	7. Air analyses. Field to be treated with chlordane. Sequence 2, preapplication			
B-7	NE corner	5/11	Impinger	ND
B-11	Center	5/11	Impinger	ND
B-17	SW corner	5/11	Impinger	3.20 ng/m^3
B-1	NW corner	5/12	Screen	0.38 total µg
B-2	NW corner	5/12	Screen	0.45 " "
B-3	NW corner	5/12	Screen	0.48 " "
B-4	NW corner	5/12	Screen	0.58 " "
B-6	NW corner	5/12	Screen	0.36 " "
B-9	SE corner	5/12	Screen	0.29 " "
B-10-B	Center	5/12	Screen	0.32 " "
B-10-C	Center	5/12	Screen	0.35 " "
B-12	NW Corner	5/12	Screen	0.39 " "
B-14	SW Corner	5/12	Screen	0.26 " "
B-15	SW Corner	5/12	Screen	0.26 " "
B-16	SW Corner	5/12	Screen	0.27 " "
	8. Air analyses. Field treated with chlordane. Sequence 3, postapplication			
B-7	NE corner	5/13	Impinger	37.9 ng/m^3
B-11	Center	5/13	Impinger	0.20 total µg
B-17	SW corner	5/13	Impinger	23.1 ng/m^3
B-2	NE corner	5/16	Screen	41.2 total µg
B-3	NE corner	5/16	Screen	36.0 " "
B-4	NE corner	5/16	Screen	23.6 " "
B-5	NE corner	5/16	Screen	29.5 " "
B-6	NE corner	5/16	Screen	28.7 " "
B-9	SE corner	5/16	Screen	20.8 " "
B-10-A	Center	5/16	Screen	11.0 " "
B-10-B	Center	5/16	Screen	12.8 " "
B-10-C	Center	5/16	Screen	19.3 " "
B-12	NW corner	5/16	Screen	14.4 " "
B-14	SW corner	5/16	Screen	17.0 " "
B-15	SW corner	5/16	Screen	14.8 " "
B-16	SW corner	5/16	Screen	18.0 " "

Tables D 1-11 (Cont.)

ANALYSES OF AMBIENT AIR AND WATER CONTAMINANTS FROM AN INDIANA CORNFIELD

Sample	Location	Date (1975)	Type	Results
	9. Air analyses. Field treated with chlordane. Sequence 4, postapplication			
B-7	NE corner	5/21	Impinger	13.7 ng/m^3
B-11	Center	5/21	Impinger	0.12 total µg
B-17	SW corner	5/21	Impinger	14.1 ng/m^3
B-2	NE corner	5/22	Screen	38.8 total µg
B-3	NE corner	5/22	Screen	25.6 " "
B-4	NE corner	5/22	Screen	7.00 " "
B-5	NE corner	5/22	Screen	36.2 " "
B-6	NE corner	5/22	Screen	43.0 " "
B-9	SE corner	5/22	Screen	22.1 " "
B-10-A	Center	5/22	Screen	23.3 " "
B-10-B	Center	5/22	Screen	33.5 " "
B-10-C	Center	5/22	Screen	29.9 " "
B-12	NW corner	5/22	Screen	28.0 " "
B-14	SW corner	5/22	Screen	6.40 " "
B-15	SW corner	5/22	Screen	7.41 " "
B-16	SW corner	5/22	Screen	8.04 " "
	10. Air analyses. Field treated with chlordane. Sequence 5, postapplication			
B-7	NE corner	6/4	Impinger	33.8 ng/m^3
B-11	Center	6/4	Impinger	0.92 total µg
B-17	SW corner	6/4	Impinger	ND
B-2	NE corner	6/5	Screen	6.10 " "
B-3	NE corner	6/5	Screen	5.50 " "
B-4	NE corner	6/5	Screen	9.20 " "
B-6	NE corner	6/5	Screen	13.2 " "
B-9	SE corner	6/5	Screen	1.80 " "
B-10-A	Center	6/5	Screen	7.52 " "
B-10-B	Center	6/5	Screen	8.20 " "
B-10-C	Center	6/5	Screen	9.10 " "
B-12	NW corner	6/5	Screen	4.10 " "
B-14	SW corner	6/5	Screen	NM†††
B-15	SW corner	6/5	Screen	NM
B-16	SW corner	6/5	Screen	NM

††† *Not measured*

Tables D 1-11 (Cont.)

ANALYSES OF AMBIENT AIR AND WATER CONTAMINANTS FROM AN INDIANA CORNFIELD

Sample	Location	Date (1975)	Insecticide - µg/l	
			Heptachlor	Chlordane
11. Water Analyses. Fields treated with heptachlor and chlordane				
Rainfall	--	5/11	ND	ND
Tile Drain	Hept. Field	5/11	ND	
Tile Drain - A	Hept. Field	5/24	ND	
Tile Drain - B	Hept. Field	5/24	ND	
Tile Drain - B	Hept. Field	5/30	2.3	
Tile Drain	Hept. Field	6/5	ND	
Watershed 15	Hept. Field	5/22	152	
Watershed 18	Hept. Field	5/22	2.2	
Watershed 19	Hept. Field	5/22	2.6	
Watershed 15	Hept. Field	5/30	7.0	
Watershed 18	Hept. Field	5/30	4.8	
Watershed 19	Hept. Field	5/30	3.8	
Watershed 5	Chlor. Field	5/22		1.6
Watershed 7	Chlor. Field	5/22		0.3
Watershed 8	Chlor. Field	5/22		0.8
Watershed 20	Chlor. Field	5/30		25

MASS SPECTROMETRY CONFIRMATION OF HEPTACHLOR AND CHLORDANE IN SAMPLES FROM THE INDIANA CORNFIELD SURVEY

Initially a water runoff sample was chosen for gas chromatography/mass spectrometry (GC/MS) identification because it contained relatively high concentrations of heptachlor and gamma chlordane. The sample was labeled Watershed 15 and represented runoff from the heptachlor field on 22 May 1975. The alumina column fraction number 1 from this sample was concentrated to 0.5 ml on a hot water bath under a gentle stream of filtered air. A 1 µl sample of the concentrated sample was injected into a Finnigan Model 1015 gas chromatograph/mass spectrometer system. The resulting mass spectrum [Fig. D-1] was a perfect match to a heptachlor standard run concurrently using the same instrumental conditions [Fig. D-2].

The impinger air samples H-7, H-11, and H-18 collected immediately after application in the heptachlor field were analyzed next. The first alumina column fraction from each of these samples was combined, concentrated, and analyzed on the GC/MS. Again the mass spectrum was a perfect match [Fig. D-3] to the standard heptachlor. These results unequivocally confirm the identity of heptachlor in the runoff water and ambient air samples taken from the heptachlor field.

A standard gamma chlordane sample was run on the GC/MS [Fig. D-4], along with the gamma chlordane in the combined alumina column fractions 3 and 4 of a technical chlordane standard [Fig. D-5]. These two spectra were identical. The alumina column fractions 3 and 4 from the H-15 runoff water sample were combined, concentrated and analyzed on the GC/MS [Fig. D-6]. The resulting spectrum was a match with the gamma chlordane standard and the gamma chlordane separated from technical chlordane by column chromatography [Figs. D-4 and D-5]. This confirmed the identity of chlordane in the water samples.

The alumina column fractions 3 and 4 from the cloth air samples H-16, H-17, B-3, B-4 and B-5 were combined and concentrated; these samples were taken from both fields immediately after application. The combined sample was analyzed on the GC/MS and the presence of gamma chlordane was quite clear [Fig. D-7]. Another set of samples from the heptachlor field (cloth samples H-2, H-3, H-4, H-5, H-9, and H-10) taken during sequence III were combined, concentrated and analyzed on the GC/MS [Fig. D-8]. This combined sample also showed clear evidence of the presence of gamma chlordane.

FIGURE D-1

SPECTRUM NUMBER 25 - 20

H-15-W FRAC NO 1 IN 0.5 ML

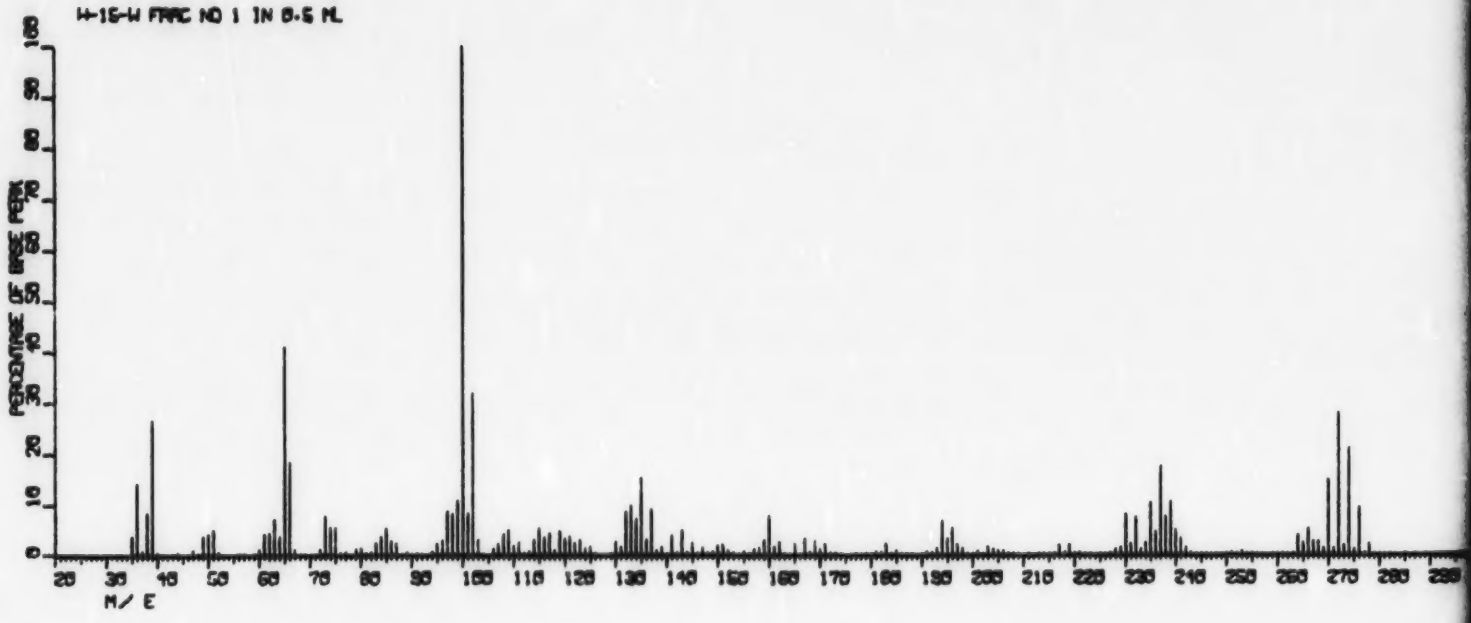
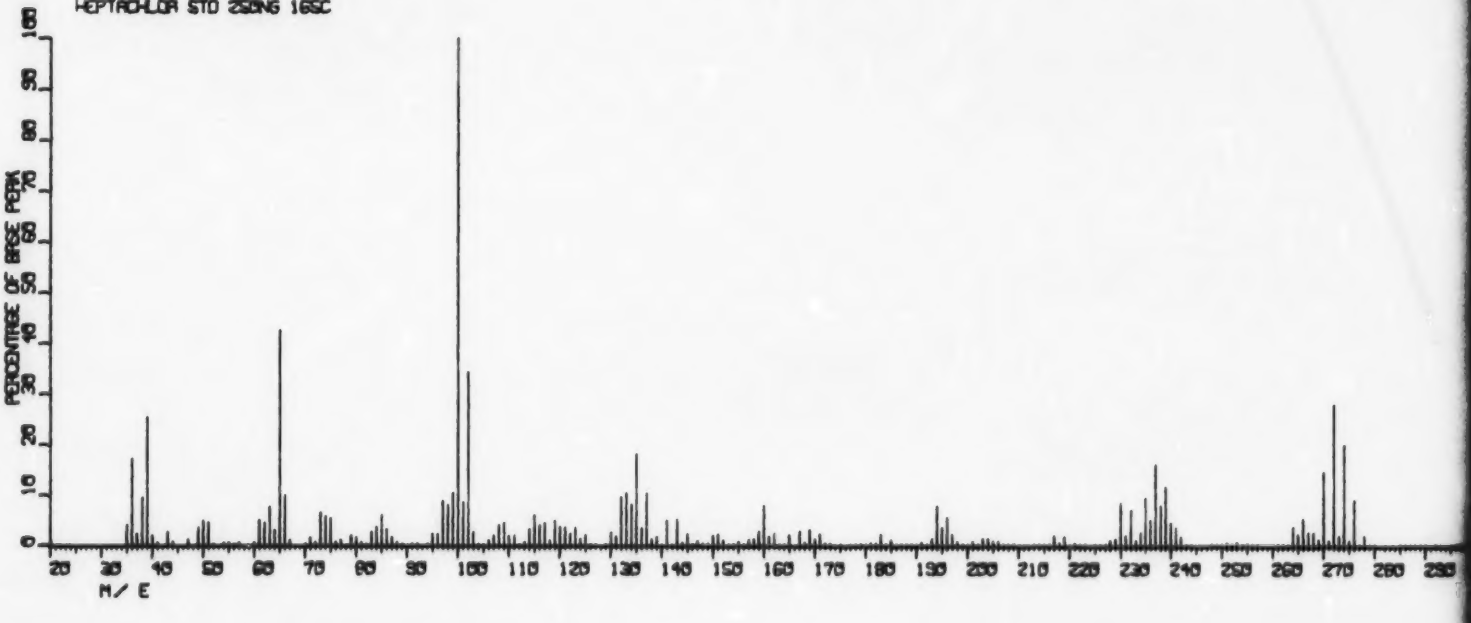


FIGURE D-2

SPECTRUM NUMBER 17 - 14

HEPTACHLOR STD 250NG 16SC



10
5
0
PERCENTAGE OF TOTAL
160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410

10
5
0
PERCENTAGE OF TOTAL
160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410

FIGURE D-3

SPECTRUM NUMBER 21 - 19

M-7-11-18 FR-1 COMB 165C 7-30-75

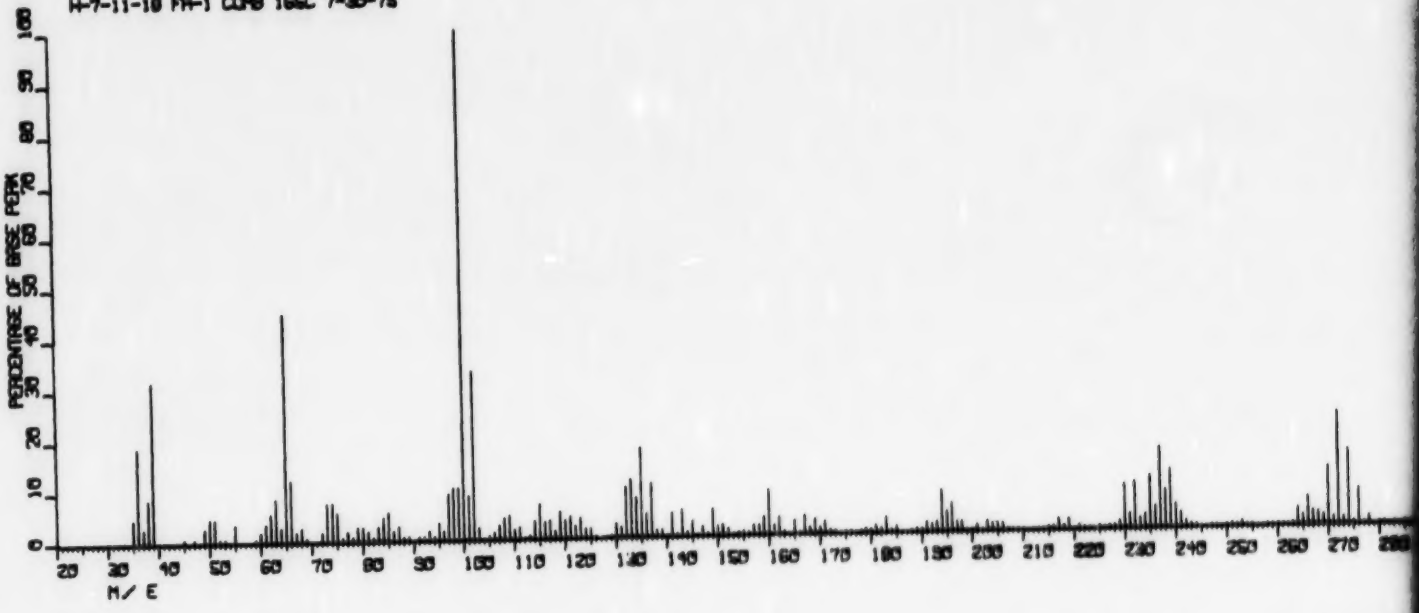
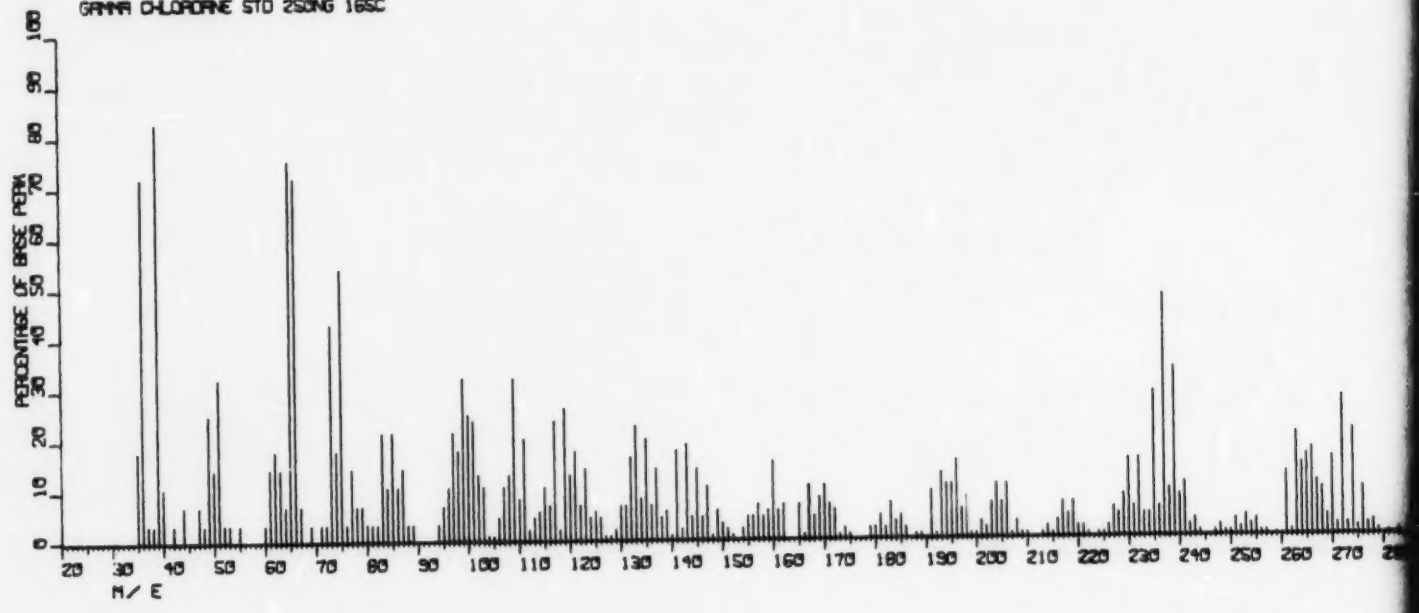
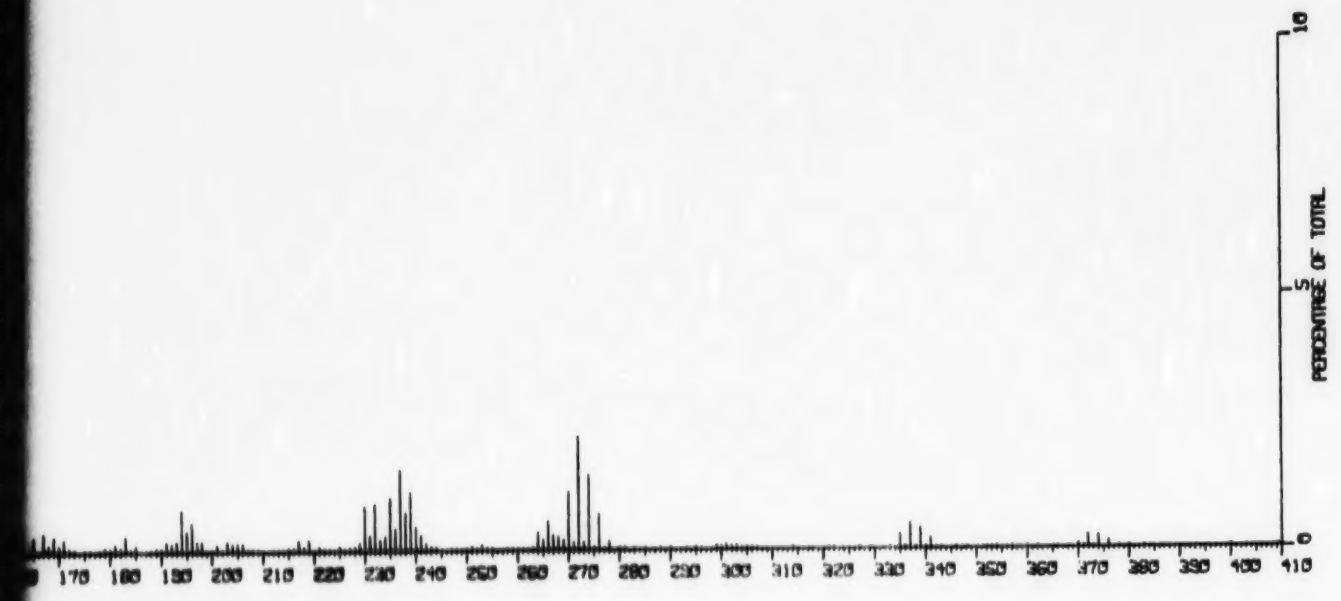


FIGURE D-4

SPECTRUM NUMBER 59 - 53

GAMMA CHLORDANE STD 2SONG 165C




10
5
0
PERCENTAGE OF TOTAL
170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410

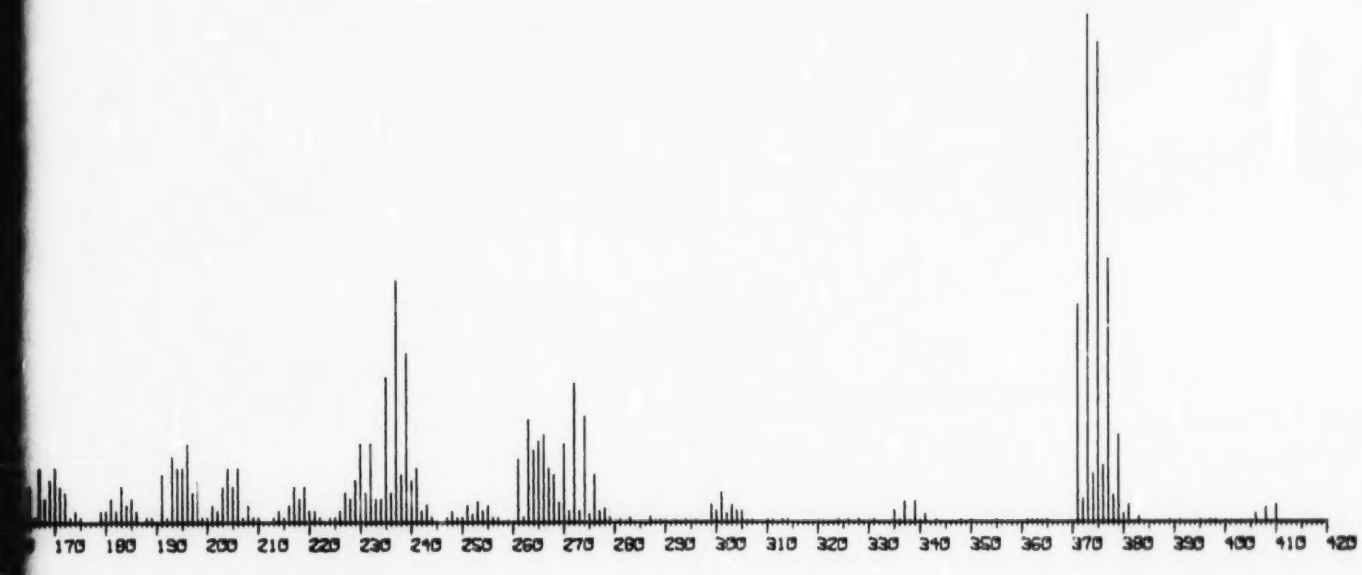

170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420

FIGURE D-5

SPECTRUM NUMBER 51 - 47

STD TECH CHLOR AL FR 3-4 250NG 16SC

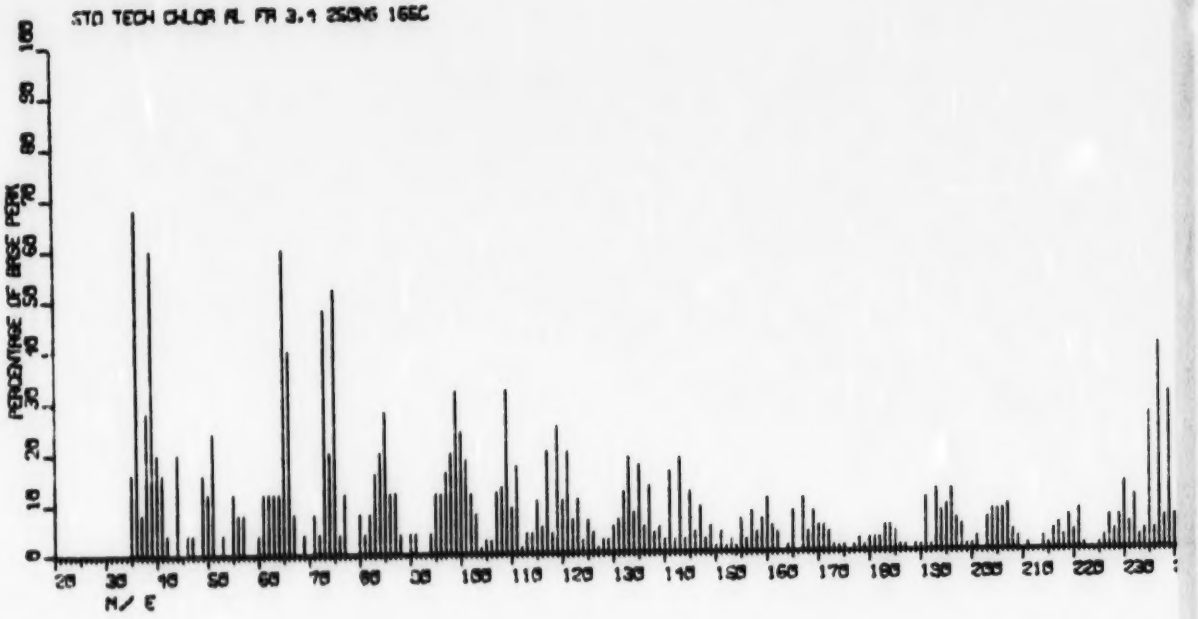
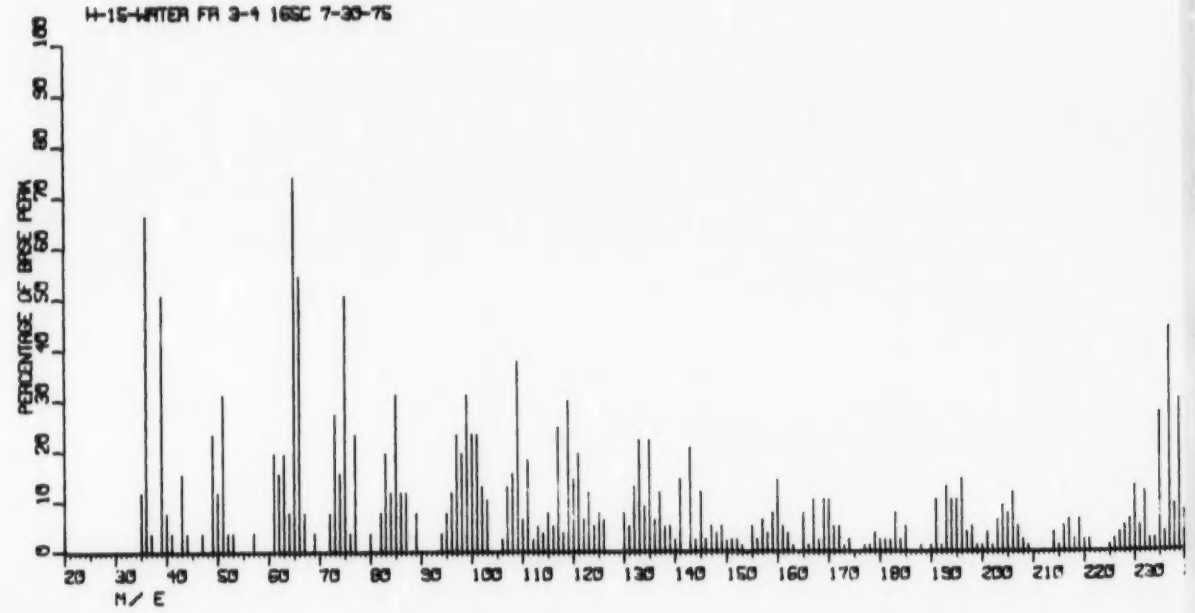
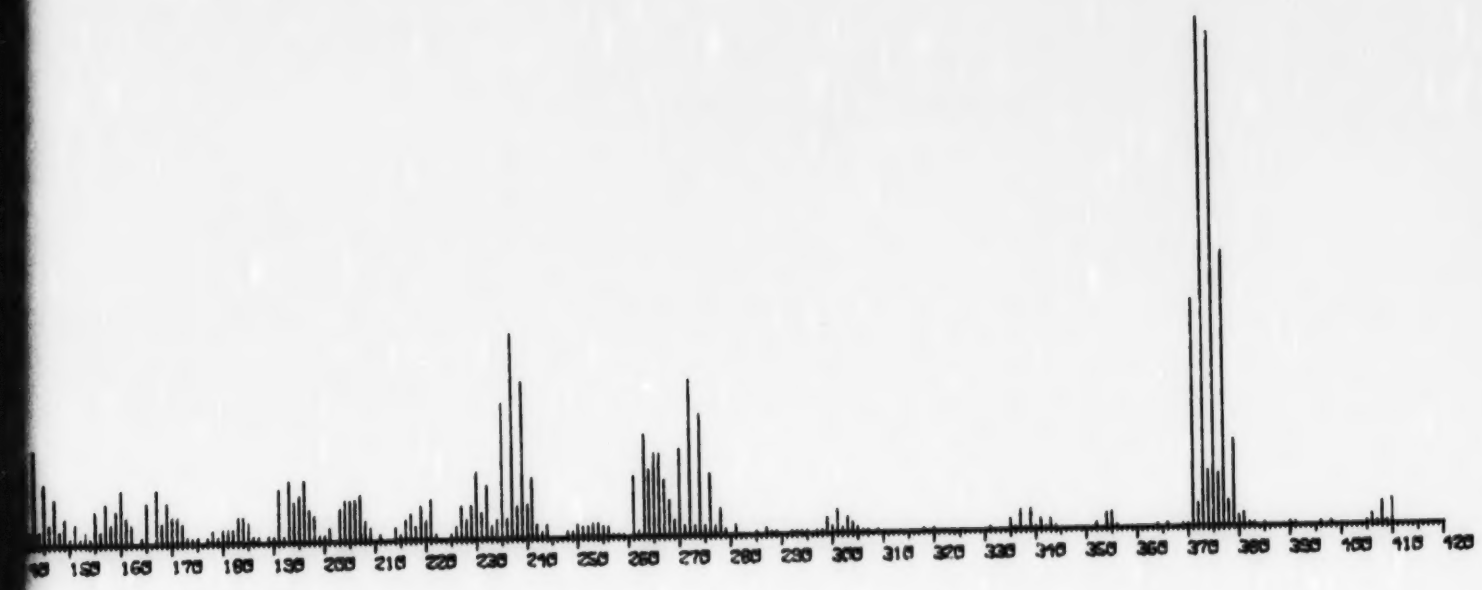


FIGURE D-6

SPECTRUM NUMBER 47 - 43

H-15-WATER FR 3-4 16SC 7-30-75

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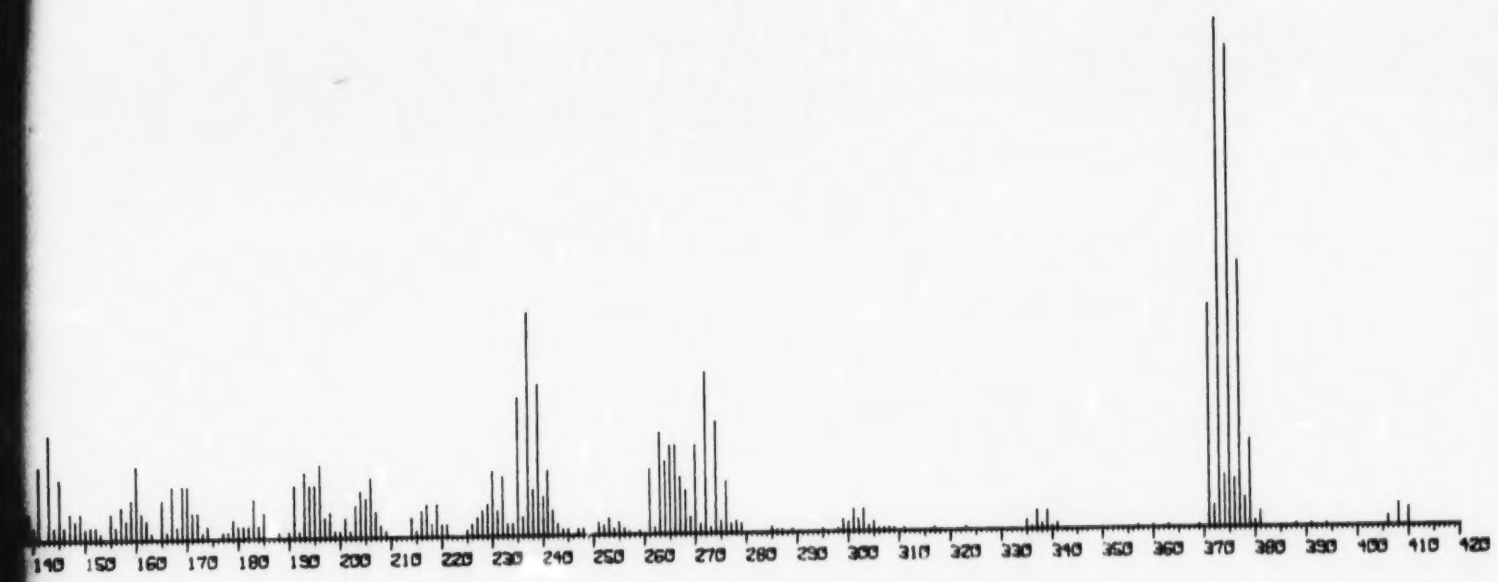

140 150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420

FIGURE D-7

SPECTRUM NUMBER 59 - 64

M-16,17-8-2,4,5, FR 2,4 COMB 7-31-75

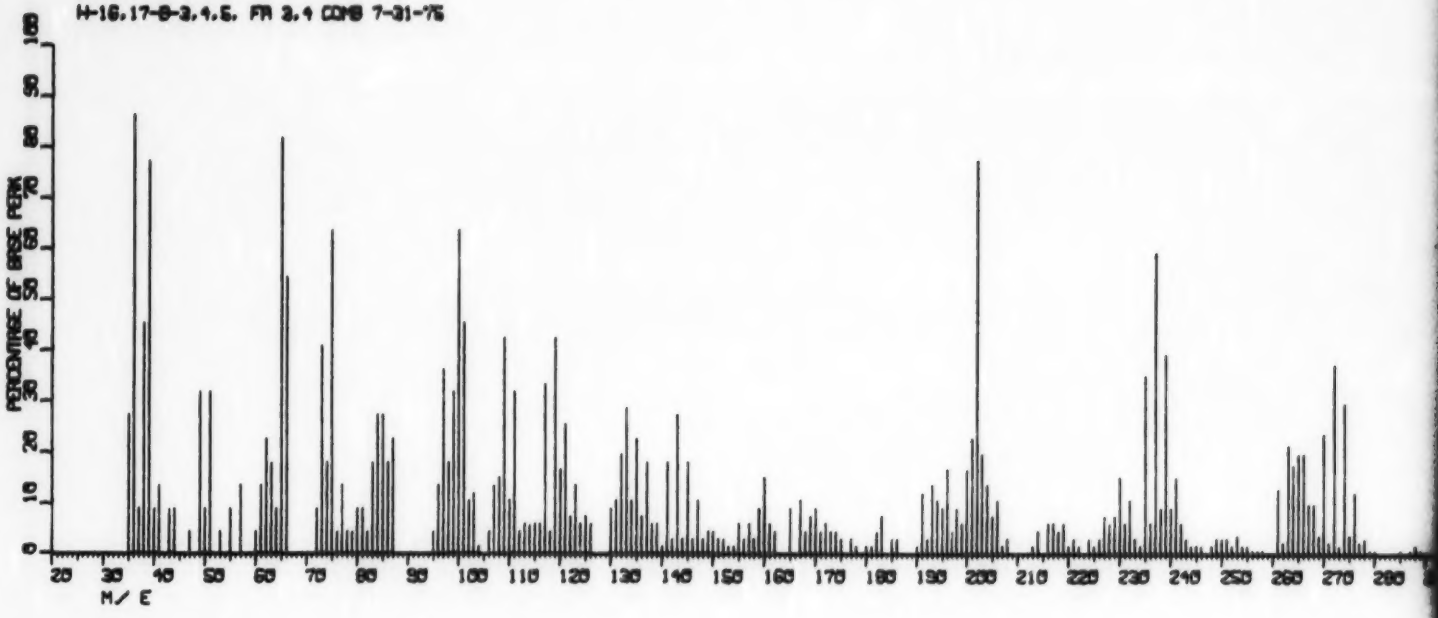
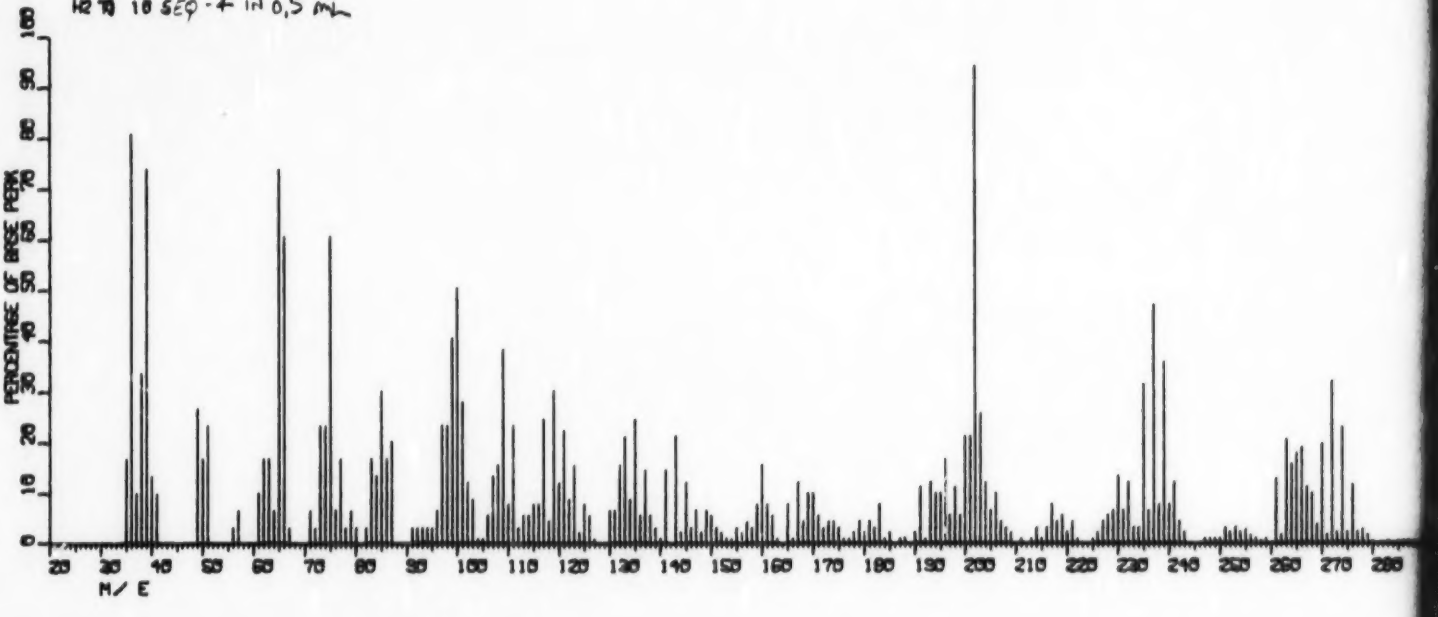
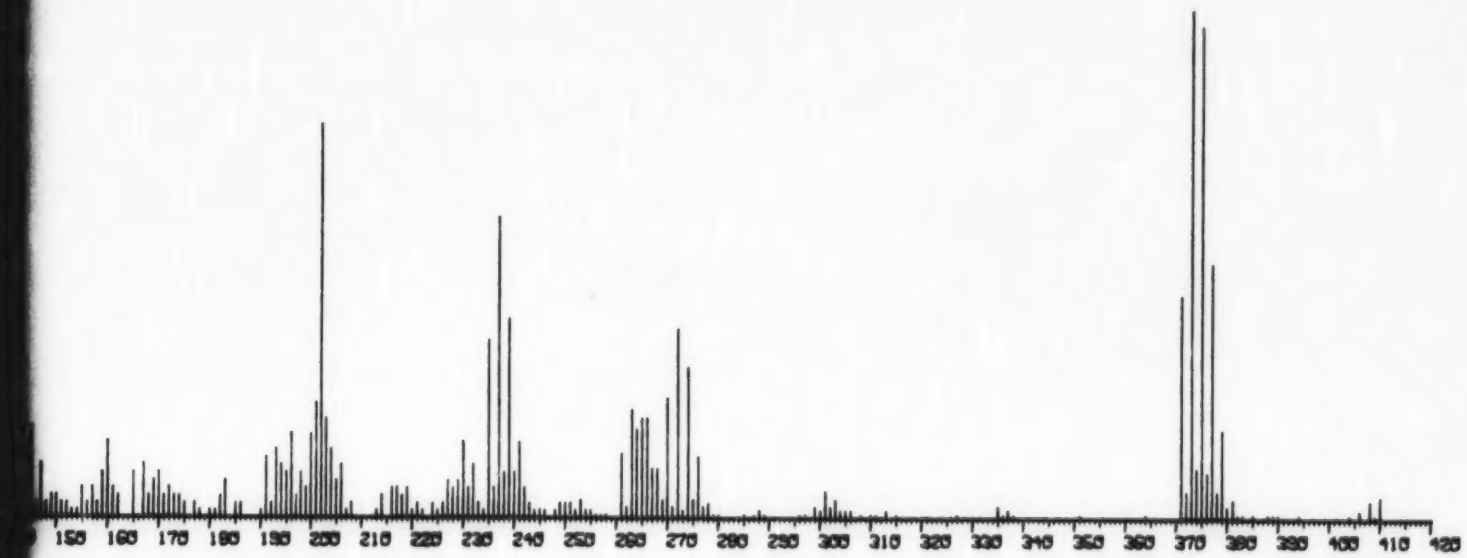


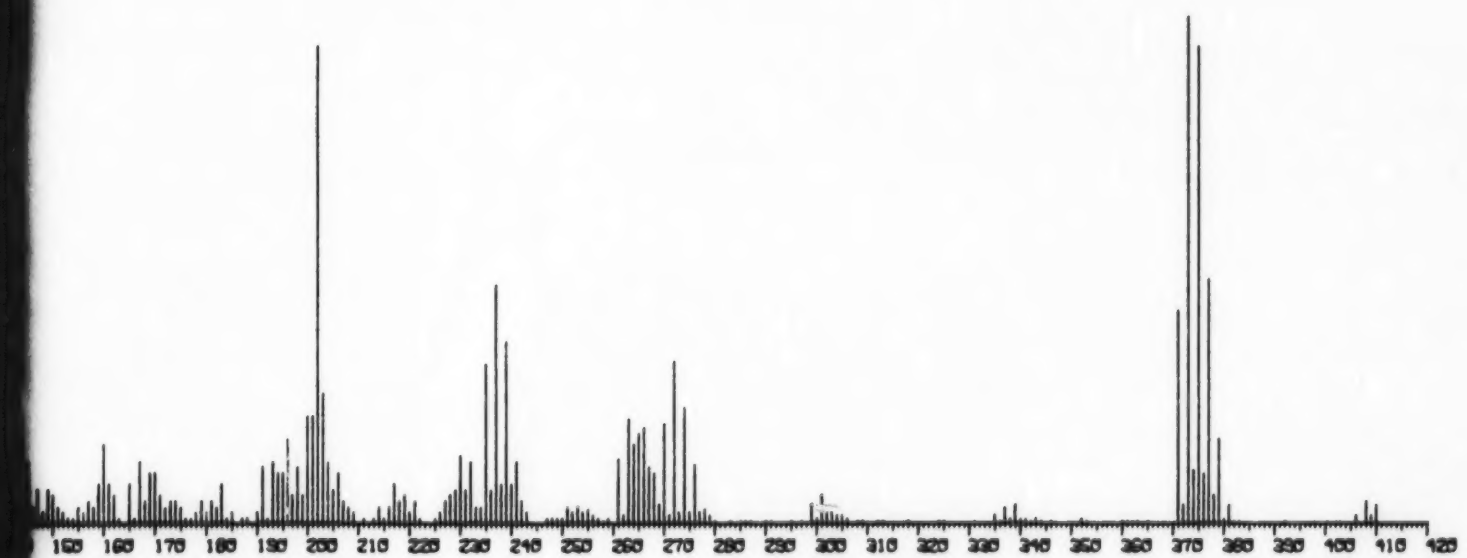
FIGURE D-8

SPECTRUM NUMBER 46 - 48

M2 78 10 SEQ - 4 IN 0,5 mL




150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420


150 160 170 180 190 200 210 220 230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390 400 410 420

APPENDIX E
CHAIN OF CUSTODY
PROCEDURES

47<

ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF ENFORCEMENT
NATIONAL FIELD INVESTIGATIONS CENTER-- DENVER
BUILDING 53, BOX 25227, DENVER FEDERAL CENTER
DENVER, COLORADO 80225

July 24, 1974

CHAIN OF CUSTODY PROCEDURES

General:

The evidence gathering portion of a survey should be characterized by the minimum number of samples required to give a fair representation of the effluent or water body from which taken. To the extent possible, the quantity of samples and sample locations will be determined prior to the survey.

Chain of Custody procedures must be followed to maintain the documentation necessary to trace sample possession from the time taken until the evidence is introduced into court. A sample is in your "custody" if:

1. It is in your actual physical possession, or

2. It is in your view, after being in your physical possession, or

3. It was in your physical possession and then you locked it up in a manner so that no one could tamper with it.

All survey participants will receive a copy of the survey study plan and will be knowledgeable of its contents prior to the survey. A pre-survey briefing will be held to re-appraise all participants of the survey objectives, sample locations and Chain of Custody procedures. After all Chain of Custody samples are collected, a de-briefing will be held in the field to determine adherence to Chain of Custody procedures and whether additional evidence type samples are required.

Sample Collection:

1. To the maximum extent achievable, as few people as possible should handle the sample.

2. Stream and effluent samples shall be obtained, using standard field sampling techniques.

3. Sample tags (Exhibit I) shall be securely attached to the sample container at the time the complete sample is collected and shall contain, at a minimum, the following information: station number, station location, date taken, time taken, type of sample, sequence number (first sample of the day - sequence No. 1, second sample - sequence No. 2, etc.), analyses required and samplers. The tags must be legibly filled out in ballpoint (waterproof ink).

Chain of Custody Procedures (Continued)

Sample Collection (Continued)

4. Blank samples shall also be taken with preservatives which will be analyzed by the laboratory to exclude the possibility of container or preservative contamination.

5. A pre-printed, bound Field Data Record logbook shall be maintained to record field measurements and other pertinent information necessary to refresh the sampler's memory in the event he later takes the stand to testify regarding his action's during the evidence gathering activity. A separate set of field notebooks shall be maintained for each survey and stored in a safe place where they could be protected and accounted for at all times. Standard formats (Exhibits II and III) have been established to minimize field entries and include the date, time, survey, type of samples taken, volume of each sample, type of analysis, sample numbers, preservatives, sample location and field measurements such as temperature, conductivity, DO, pH, flow and any other pertinent information or observations. The entries shall be signed by the field sampler. The preparation and conservation of the field logbooks during the survey will be the responsibility of the survey coordinator. Once the survey is complete, field logs will be retained by the survey coordinator, or his designated representative, as a part of the permanent record.

6. The field sampler is responsible for the care and custody of the samples collected until properly dispatched to the receiving laboratory or turned over to an assigned custodian. He must assure that each container is in his physical possession or in his view at all times, or locked in such a place and manner that no one can tamper with it.

7. Colored slides or photographs should be taken which would visually show the outfall sample location and any water pollution to substantiate any conclusions of the investigation. Written documentation on the back of the photo should include the signature of the photographer, time, date and site location. Photographs of this nature, which may be used as evidence, shall also be handled recognizing Chain of Custody procedures to prevent alteration.

Transfer of Custody and Shipment:

1. Samples will be accompanied by a Chain of Custody Record which includes the name of the survey, samplers signatures, station number, station location, date, time, type of sample, sequence number, number of containers and analyses required (Fig. IV). When turning over the possession of samples, the transferor and transferee will sign, date and time the sheet. This record sheet

Chain of Custody Procedures (Continued)

allows transfer of custody of a group of samples in the field, to the mobile laboratory or when samples are dispatched to the NFIC - Denver laboratory. When transferring a portion of the samples identified on the sheet to the field mobile laboratory, the individual samples must be noted in the column with the signature of the person relinquishing the samples. The field laboratory person receiving the samples will acknowledge receipt by signing in the appropriate column.

2. The field custodian or field sampler, if a custodian has not been assigned, will have the responsibility of properly packaging and dispatching samples to the proper laboratory for analysis. The "Dispatch" portion of the Chain of Custody Record shall be properly filled out, dated, and signed.

3. Samples will be properly packed in shipment containers such as ice chests, to avoid breakage. The shipping containers will be padlocked for shipment to the receiving laboratory.

4. All packages will be accompanied by the Chain of Custody Record showing identification of the contents. The original will accompany the shipment, and a copy will be retained by the survey coordinator.

5. If sent by mail, register the package with return receipt requested. If sent by common carrier, a Government Bill of Lading should be obtained. Receipts from post offices and bills of lading will be retained as part of the permanent Chain of Custody documentation.

6. If samples are delivered to the laboratory when appropriate personnel are not there to receive them, the samples must be locked in a designated area within the laboratory in a manner so that no one can tamper with them. The same person must then return to the laboratory and unlock the samples and deliver custody to the appropriate custodian.

Laboratory Custody Procedures:

1. The laboratory shall designate a "sample custodian." An alternate will be designated in his absence. In addition, the laboratory shall set aside a "sample storage security area." This should be a clean, dry, isolated room which can be securely locked from the outside.

2. All samples should be handled by the minimum possible number of persons.

3. All incoming samples shall be received only by the custodian, who will indicate receipt by signing the Chain of Custody Record Sheet

accompanying the samples and retaining the sheet as permanent records. Couriers picking up samples at the airport, post office, etc. shall sign jointly with the laboratory custodian.

4. Immediately upon receipt, the custodian will place the sample in the sample room, which will be locked at all times except when samples are removed or replaced by the custodian. To the maximum extent possible, only the custodian should be permitted in the sample room.

5. The custodian shall ensure that heat-sensitive or light-sensitive samples, or other sample materials having unusual physical characteristics, or requiring special handling, are properly stored and maintained.

6. Only the custodian will distribute samples to personnel who are to perform tests.

7. The analyst will record in his laboratory notebook or analytical worksheet, identifying information describing the sample, the procedures performed and the results of the testing. The notes shall be dated and indicate who performed the tests. The notes shall be retained as a permanent record in the laboratory and should note any abnormalities which occurred during the testing procedure. In the event that the person who performed the tests is not available as a witness at time of trial, the government may be able to introduce the notes in evidence under the Federal Business Records Act.

8. Standard methods of laboratory analyses shall be used as described in the "Guidelines Establishing Test Procedures for Analysis of Pollutants," 38 F.R. 28758, October 16, 1973. If laboratory personnel deviate from standard procedures, they should be prepared to justify their decision during cross-examination.

9. Laboratory personnel are responsible for the care and custody of the sample once it is handed over to them and should be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the tests were run.

10. Once the sample testing is completed, the unused portion of the sample together with all identifying tags and laboratory records, should be returned to the custodian. The returned tagged sample will be retained in the sample room until it is required for trial. Strip charts and other documentation of work will also be turned over to the custodian.

Chain of Custody Procedures (Continued)

11. Samples, tags and laboratory records of tests may be destroyed only upon the order of the laboratory director, who will first confer with the Chief, Enforcement Specialist Office, to make certain that the information is no longer required or the samples have deteriorated.

EXHIBIT I

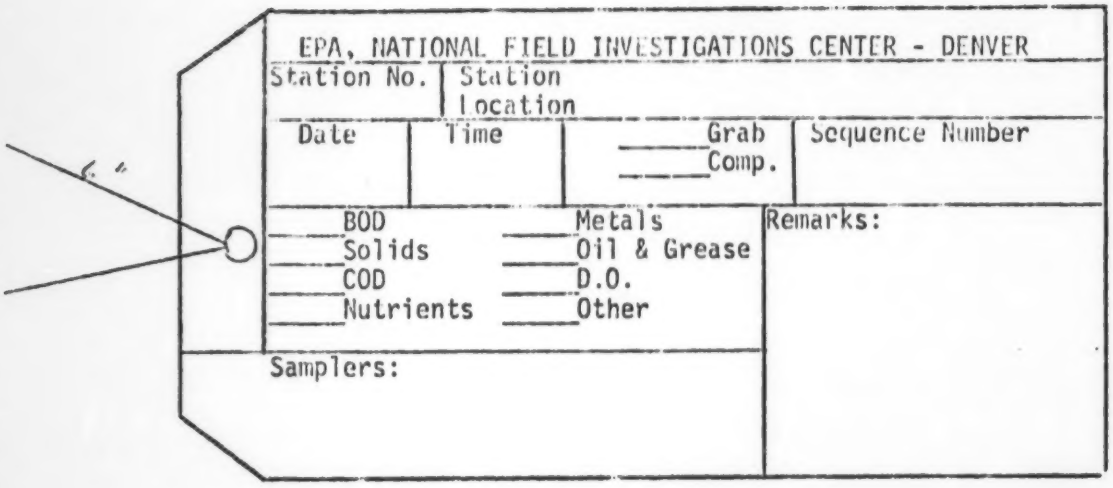


EXHIBIT II

FOR _____ SURVEY, PHASE_____, DATE _____

TYPE OF SAMPLE_____

ANALYSES REQUIRED

STATION NUMBER	STATION DESCRIPTION	TOTAL VOLUME	TYPE CONTAINER	PRESERVATIVE	NUTRIENTS	BOD	COD	TOC	TOTAL SOLIDS	SUSPENDED SOLIDS	ALKALINITY	DO	pH*	CONDUCTIVITY*	TEMPERATURE*	TOTAL COLIFORM	FECAL COLIFORM	TURBIDITY	OIL AND GREASE	METALS	BACTI	PESTICIDES	HERB	TRACE ORGANICS	PHENOL	CYANIDE

REMARKS

EXHIBIT III

Samplers: _____

_____

_____

_____

FIELD DATA RECORD

STATION	NUMBER	DATE	TIME	TEMPERATURE °C	CONDUCTIVITY µmhos/cm	pH S.U.	D.O. mg/l	Gage Ht. or Flow Ft. or CFS

EXHIBIT IV

ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF ENFORCEMENT
NATIONAL FIELD INVESTIGATIONS CENTER - DENVER
Building 53, Box 25227, Denver Federal Center
Denver, Colorado 80225

CHAIN OF CUSTODY RECORD

SURVEY | SAMPLERS: (Signature)

Station Number	Station Location	Date Time	Sample Type		Seq. #	No. of Containers	Analysis Required
			Comp.	Grab			

Relinquished by:	Received by:	Date	Time
Relinquished by:	Received by:	Date	Time
Relinquished by:	Received by Mobile Laboratory for Field Analysis:	Date	Time
Dispatched by: (Signature) Date Time	Received for Laboratory by:	Date	Time

Method of Shipment:

Distribution: Original - Accompany shipment
1 copy - Survey Coordinator Field Files